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AN INSTRUMENT FOR MEASURING THE THERMAL CONDUCTANCE OF HIGH-TEMPERATURE STRUCTURAL MATERIALS

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AF MATERIALS LABORATORY
RESEARCH AND TECHNOLOGY DIVISION
AIR FORCE SYSTEMS COMMAND
WRIGHT-PATTERSON AIR FORCE BASE, OHIO

Project 7381, Task 738103

(Prepared under Contract No. AF 33(616)-8099 by the
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FOREWORD

This study was initiated by the Applications Laboratory of the Directorate of Materials and Processes, Aeronautical Systems Division, Wright-Patterson Air Force Base, Ohio; the apparatus described was designed, constructed, and installed by Dynatech Corporation of Cambridge 39, Massachusetts, under Contract No. AF 33(616)-8099. Mr. James K. Sparrell, Manager of the Instruments Division, was the project manager, and Mr. Karl G. Coumou was the project engineer for Dynatech Corporation. 1/Lt. M. L. Minges of the Materials Engineering Branch was the monitor for the Applications Laboratory. The work was performed in support of Project No. 7381, "Materials Application," and Task No. 738103, "Data Collection and Correlation." The program has been in progress from March 1, 1961, to the present.

As part of this contract Dynatech Corporation was to have conducted a series of ten checkout and calibration tests, eight of which have been successfully completed. Several failures of the main heater as supplied to Dynatech under subcontract by Lexington Laboratories, Cambridge, Massachusetts, have necessitated a heater research and development program by Dynatech in order to fulfill the requirements of this contract. A wealth of technical information has been generated during this development program and is presented in Section 3 of this report.

This program is currently being conducted and the results of this program together with the results of the remaining tests will be described in a supplemental report.

Dynatech Corporation would like to extend its appreciation to the Rohr Aircraft Corporation, Chula Vista, California, for the provision of all honeycomb test samples, to Corning Glass Works, Corning, New York, for supplying the test samples used in the Fused Silica Multiform test and to the Republic Steel Corporation of Cleveland, Ohio, for providing the 17-4 PH precipitation hardening stainless steel test specimen.

This report is cataloged by Dynatech Corporation as Report No. 372.

ABSTRACT

A thermal conductivity apparatus employing the absolute guarded hot plate principle has been designed, developed and fabricated for the United States Air Force. The primary objective of this instrument is to test honeycomb structures at elevated temperatures. A secondary objective is to test super-alloys, ceramics, and cermets.

Operational requirements are as follows:

Sample temperature: approximately 300^{o} to $3000^{o}F$

Heat dissipation from main and guard heaters: 1000 - 20,000 Btu/hr-ft^2

Heat dissipation from auxiliary heaters: 0 - 15,000 Btu/hr-ft^2

Environments: Air, argon, vacuum approximately 10^{-5} mm Hg

Sample size: 12 in x 12 in square, 3/8 in to 2-1/2 in thick

Accuracy: $1800^{o}F$ within $\pm$ 5%
$3000^{o}F$ within $\pm$ 10%

In general these operational requirements have been met with calibration runs on copper to $1800^{o}F$ and Al_2O_3 to $3000^{o}F$ falling within the prescribed accuracy limits. Further thermal conductivity tests have been completed on René 41 Honeycomb, Titanium Alloy Honeycomb, L605 Cobalt Alloy Honeycomb, Stainless Steel, René 41, and Fused Silica. Thermal conductance tests on Tungsten and Columbium are planned. A detailed discussion is made of instrumentation and materials compatability problems encountered during the testing program.

This technical documentary report has been reviewed and is approved.

W. P. Conrardy
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Applications Laboratory
Directorate of Materials and Processes

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Section 1

INTRODUCTION

The advancement in modern aircraft development necessitates a detailed knowledge of the behavior of aircraft structural materials at high temperatures. The apparatus as described in this report has been designed to measure the thermal conductance of these materials to a maximum sample temperature of 3000°F. The device has the capability of measuring a variety of materials, whose conductances may range from 1 to 1000 Btu/hr-ft^2-°F.

During that portion of the test program completed to date, the thermal conductance has been determined at several temperatures up to 1800°F of a 2-1/2 in thick copper sample with the results well within 5% of accepted reference values. On the other hand, a 5/8 in-thick sample of aluminum oxide has been tested to 3000°F with results deviating less than 10% from the accepted reference conductivity.

The primary objective for this instrument is the testing of metallic materials in a sandwich configuration, and several measurements were made on honeycomb panels from 300°F to about 2300°F, the results of which compared favorably with values obtained analytically.

The design enables continuous testing from approximately 300° to 3000°F sample temperature, if required, without the necessity of changing the test configuration throughout the temperature range. Measurements at, or slightly higher than, room temperature can be made by rearranging the test configuration.

The conductance of cellular materials is affected by the presence of air inside the cells. Therefore, an environmental system is provided to conduct measurements in a vacuum as well. The environmental chamber can be backfilled with an inert gas if it is necessary to protect samples from oxidizing.

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Section 2

APPARATUS DESCRIPTION

The instrument may be divided into three subunits: the test section, the environmental system, and the power, control, and instrumentation system, each of which will be discussed in turn. Physically there are three pieces of equipment interconnected by electrical wiring only. The over-all dimensions are 116 inches maximum height, 105 inches maximum width, and 35 inches maximum depth. The actual configuration is shown in Figure 1.

2.1 Test Section

Following is a description of the test section, first, in over-all configuration, briefly describing the components and their relationship to each other, and second, in detail describing each of the test section components.

The test section consists of a composite stack as shown schematically in Figure 2 and has the dimensions of approximately one cubic foot. Heat generated in the 4 in x 4 in main heater flows in approximately equal quantities in the direction of the two water-cooled, copper heat sinks. In doing so, the heat passes in turn through the test samples, the ratio elements, insulation (optional), auxiliary heaters, and insulation (optional). Surrounding the main heater, to ensure unidirectional heat flow through the sample, are eight automatically-controlled guard heaters.

This is in general the test stack configuration; however, for many tests some of the components (e.g., insulation, ratio elements, auxiliary heaters) may be omitted.

2.1.1 Main and Guard Heaters

The initial high temperature heating element was constructed for Dynatech by Lexington Laboratories, Inc., Cambridge, Massachusetts, under subcontract. During this program this heater has undergone substantial modifications as a result of failures occurring during the test program. These modifications and the heater failures which necessitated them are described in detail in Section 3.1. The basic over-all configuration of the main and guard heaters has not been modified during this program.

The over-all configuration of the main and guard heater assembly is a square, flat plate, 12 in x 12 in x 3/4 in. A square arrangement was selected because of the difficulties that would be encountered in winding a circular high temperature heating element. The heater consists of nine 4 in x 4 in x 3/4 in. subassemblies, the center one serving as the main heater, which is surrounded by the eight remaining heaters serving as guard heaters. Figure 3 shows the main and guard heaters.

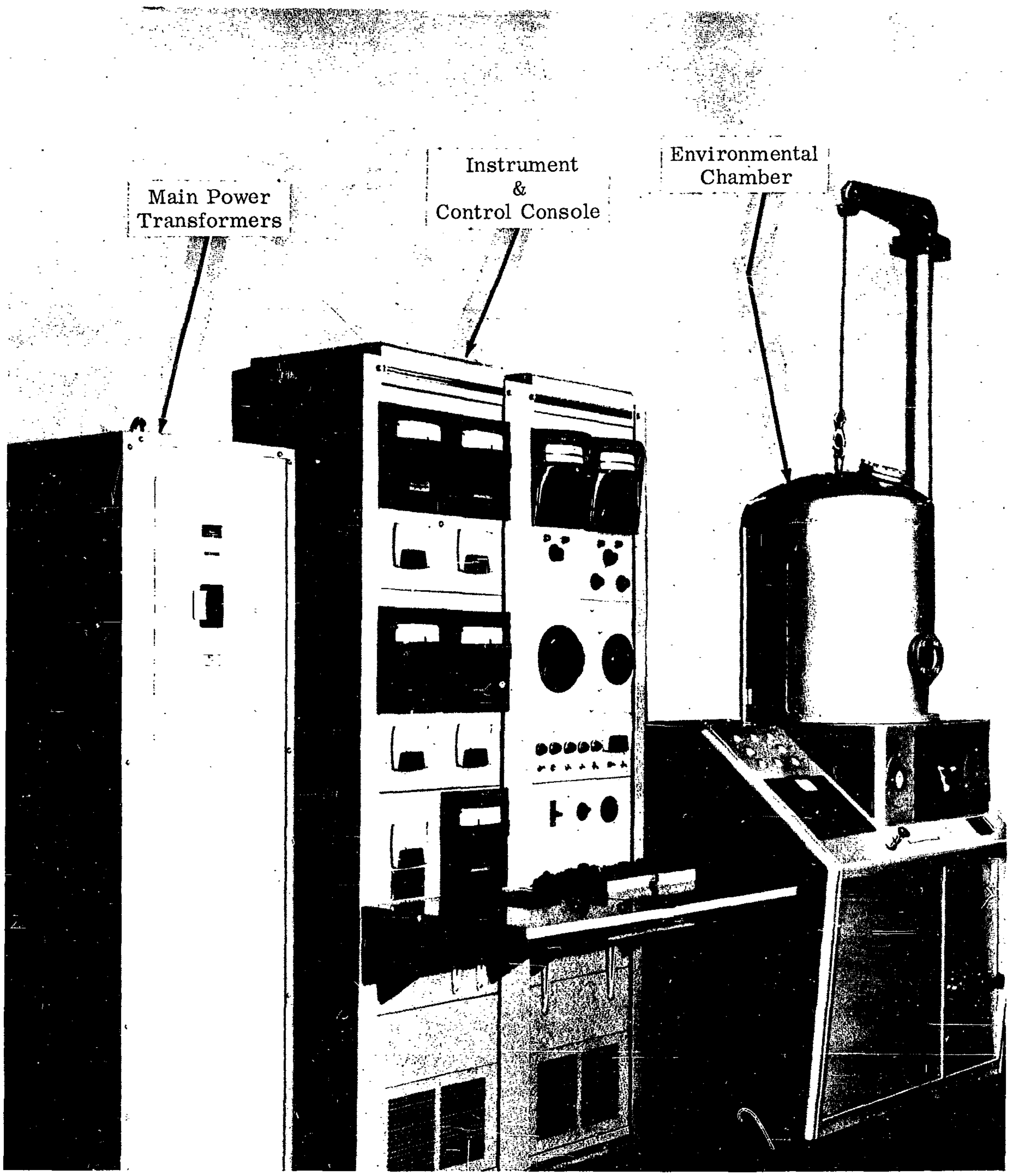


Figure 1. High Temperature Thermal Conductivity Instrument

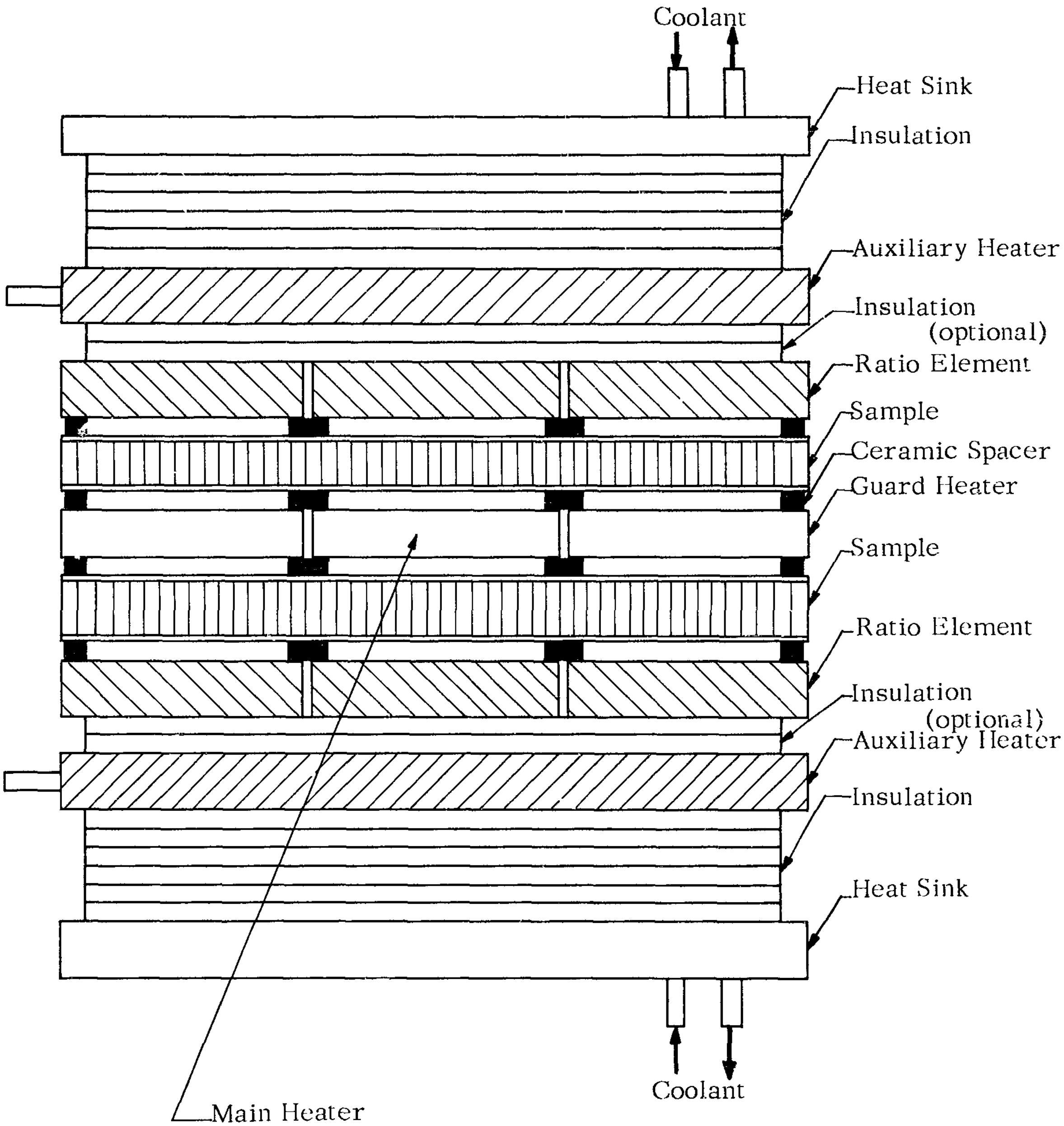


Figure 2. Typical Test Stack Employing Two Samples for Thermal Conductivity Measurement

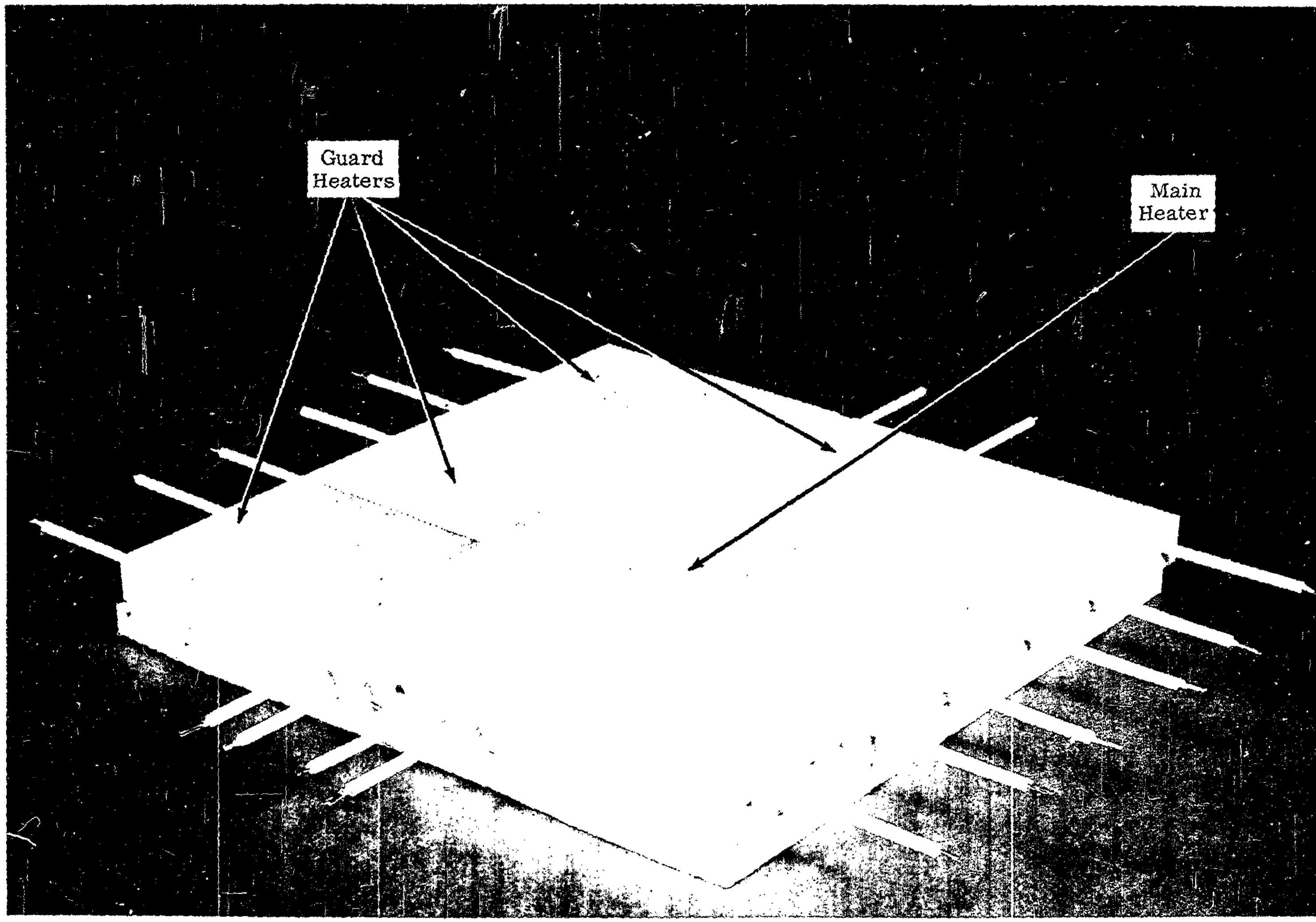


Figure 3. Main and Guard Heater Configuration

Each of the nine heaters is a grooved pure alumina block into which a platinum-rhodium resistance wire is wound. A pure alumina slip was then used to fill the grooves covering the resistance wire. A further description, including a discussion of fabrication, is presented in Section 3.1.

2.1.2 Samples

Usually two samples are used in the stack during a test, one immediately above, and one immediately below, the main and guard heaters. This configuration is in accordance with ASTM Specification C-177-45 (Ref. 1) and avoids the use of another guard heater which would otherwise be required. With solid materials, the use of identical slabs on each side of the heater provides two independent measurements of material conductivity. Unequal heat fluxes are determined by the ratio elements (described in the following section). With cellular samples such as honeycomb structures, the effect of internal convection, as influenced by heat flux direction relative to gravity, may be evaluated.

An alternate configuration that may be used at lower temperatures (1800°F max) is shown in Figure 4. In this configuration, a single sample is placed on one side of the main and guard heater and a differential slab is placed on the other side. In this configuration, one of the auxiliary heaters is used to buck the main heater, forcing all of the heat from the main heater through the single sample. A differential thermocouple wrapped around the differential slab indicates an adiabatic condition with a null signal. Because of the high heat flux required this test configuration was employed during the copper calibration test.

A third test configuration, similar to the first described, may be used with samples of different materials. With this arrangement the two different samples are placed on either side of the main and guard heaters and the ratio elements are used to establish the ratio of heat fluxes through the two samples.

The sample size is 12 in by 12 in square with a thickness depending on the thermal conductivity range of the specific material or honeycomb structure being tested. A sample of sufficient thickness to produce at least a 30°F temperature difference across the sample at maximum heat flux is used. This temperature drop is required to meet ASTM specifications on the guard heater control with the selected instrumentation (sensors and controls).

Either a single 12 in x 12 in slab or nine 4 in x 4 in blocks may be used as a sample. Both sample configurations have proved successful. With high conductivity materials such as the copper sample, the guarding problem is eased somewhat by using nine 4 in x 4 in samples and separating them slightly with insulation inserts.

2.1.3 Ratio Elements

As the name suggests, the ratio elements serve to determine the ratio of the heat flux supplied from the main heater that passes in each direction through the samples. Although the basic ASTM Specification C-177-45, upon which this instrument's design was based, does not use ratio elements, these were added to

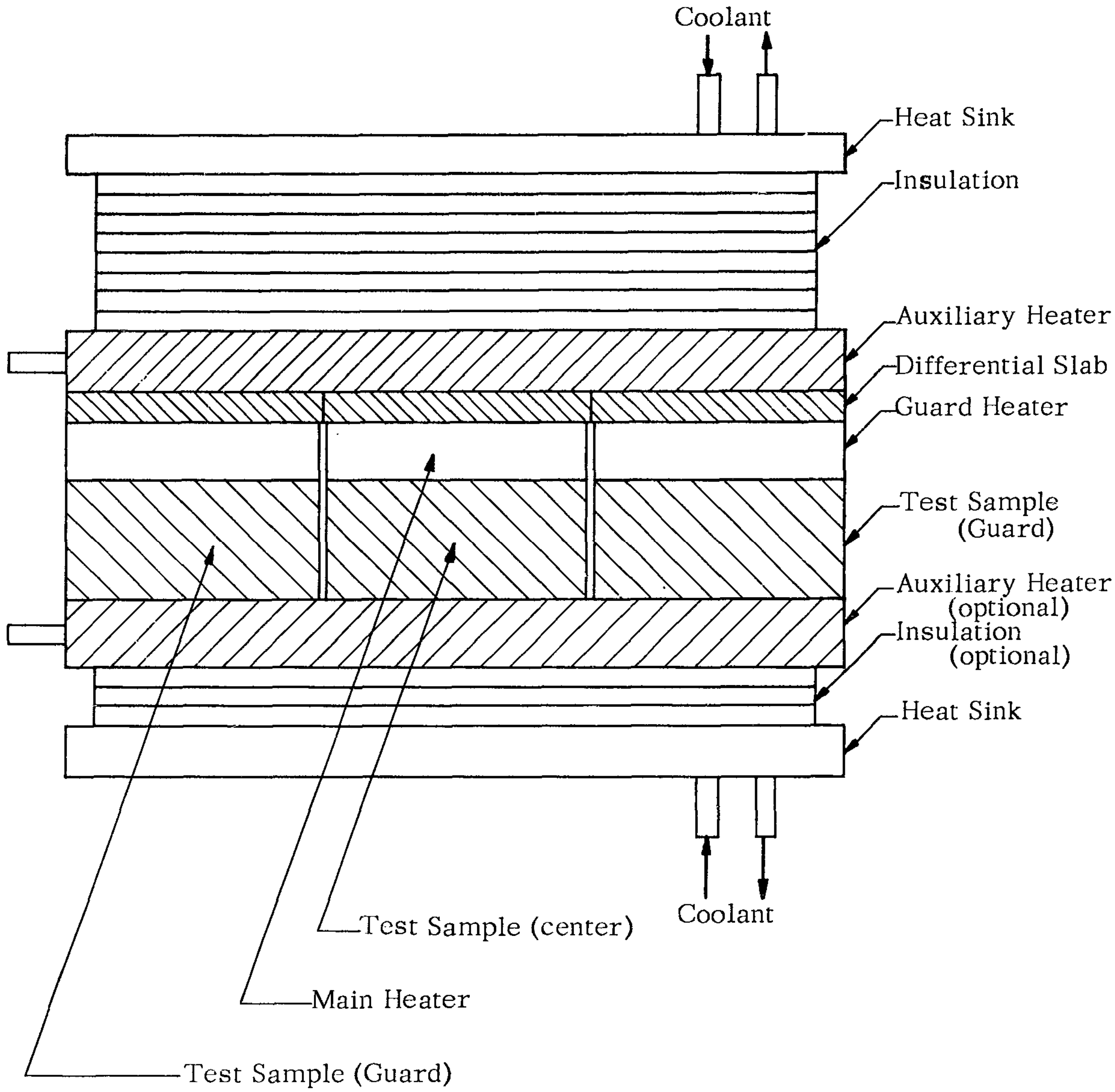


Figure 4. Typical Test Stack Employing a Single Sample for Thermal Conductivity Measurement

enhance the instrument's capabilities. Being able to determine the ratio of heat flux through the two specimens permits the testing of two different samples, or in the case of cellular or honeycomb structures, permits an evaluation of heat transfer due to internal convection.

The ratio elements consist of identical ceramic slabs which are placed adjacent to the cold faces of the two samples. Each ratio element is made up of nine 4 in x 4 in slabs forming a 12 in x 12 in slab in place. Thermocouples installed in the two surfaces of each center slab provide the temperature differences which in turn are proportional to the heat flux through each ratio element. The ratio of the temperature differences obtained from the two ratio elements is equal to the ratio of heat fluxes through the two ratio elements.

Three sets of ratio elements were provided with the instrument. It was deemed important to select materials having a linear-thermal-conductivity-versus-temperature characteristic to ensure equal average values for the conductivity of both ratio elements with different temperature differences across each ratio element. The ratio of heat fluxes and temperature distributions in the test stack are controlled during the tests to ensure that the average temperatures of the two ratio elements are equal.

The materials for the three sets of ratio elements were selected to have conductivity values which would restrict the ratio element temperature drops to between 50°F and 300°F over the entire heat flux range of the instrument. Alumina was selected for use at highest heat fluxes, silicon carbide for intermediate heat fluxes, and porous mullite for lowest heat fluxes.

2.1.4 Auxiliary Heaters

The auxiliary heaters are incorporated in the stack design to serve as a variable thermal resistance and to provide a means of balancing the mean temperature of the ratio elements.

It would be extremely inconvenient during a test run to disassemble the stack because additional insulation is required between the sample and the heat sink, especially where tests are conducted within the environmental chamber. The auxiliary heaters provide a means of adjusting the average sample temperature independent of the heat flux. If tests were conducted without auxiliary heaters on a sample over a large temperature range, the heat flux delivered by the main heater would have to be increased to achieve an increase in sample temperature. This would result in a small sample temperature difference at low temperatures, and a large difference at high temperatures. With the auxiliary heater, a test may be conducted at constant heat flux over a large sample temperature range.

The power to one of the auxiliary heaters is manually set to achieve the desired sample temperature. The other heater is automatically controlled to maintain equal average temperatures of the ratio elements. The auxiliary heaters are designed to yield a maximum power dissipation of 15,000 Btu/hr-ft^2, and they are capable of maximum operating temperatures of 2200°F. The design employs KANTHAL DA-ribbon cast in an Alundum cement, especially developed to match the expansion coefficient of KANTHAL alloy. The casting has been machined to the

approximate dimensions 12 in x 12 in x 1 in and is contained in a specially designed Inconel sheet-metal box to facilitate handling.

2.1.5 Insulating Slabs

In order to reach sample temperatures of 3000°F and still use tap water for heat sink cooling, some insulation must be placed between the sample and the heat sink. Depending on the operating heat flux and the required sample temperature, the thickness of this insulation varies. For this reason it was decided to use fairly thin (1/8 in) ceramic slabs. The material was carefully selected so that when the maximum required amount of insulation is installed, the total thickness of the insulation on each side of the stack would be approximately 1 in (8 slabs). Wesgo VX super-refractory was selected on the basis of its conductivity, its low coefficient of thermal expansion, and its strength.

A thermal gradient across a flat plate of material will cause the plate to assume a bowed shape with a radius of curvature inversely proportional to the temperature drop and the coefficient of thermal expansion, and directly proportional to the material thickness. This bowing can cause nonuniform thermal contact resistance, thus imposing a difficult balancing task on the guard heaters, and probably resulting in a severe deviation of the unidirectional heat flow pattern through the stack. The low coefficient of thermal expansion for the VX-slabs is compatible with our aim to keep the bowing to a minimum. Moreover, during testing, the stack is physically compressed to eliminate as much of the bowing as possible. Again, the 1/8 in slab thickness (small temperature drop per slab), low expansion coefficient, and relatively high flexibility and tensile strength for VX super-refractory keep the slabs from failing under the compressive load.

In operation the insulation may actually be divided and placed on both sides of the auxiliary heaters. In high temperature runs this becomes necessary since the auxiliary heaters are limited to a maximum operating temperature of 2200°F.

2.1.6 Heat Sinks

The heat sinks are essentially copper slabs with multiple water passages machined internally to maintain a constant surface temperature. The over-all dimensions are 12 in x 12 in x 1/2 in. They are designed for a maximum power dissipation of 35,000 Btu/hr (maximum main-plus-guard-heater output plus maximum-auxiliary-heater output). With a water flow of four gallons per minute, the total temperature increase of the cooling water under the worst conditions is 17°F. A constant surface temperature becomes especially important at minimum sample temperature runs. For minimum temperature, all insulation, auxiliary heaters, and even the ratio elements can be removed.

Hydraulic quick-connectors are used between the heat sink inlet and outlet and the water feed-throughs mounted in the base plate of the environmental system. They have been proved to be vacuum tight to at least 1×10^{-5} mm Hg.

2.1.7 Circumferential Insulation

To minimize radial heat losses and prevent an overload of the guard heaters, insulation is provided around the stack. Two approaches have been taken and both worked satisfactorily. The application of insulation is quite a delicate job in view of the many heater leads and thermocouples that exit from the stack (Figure 5). It was found that rolled-up sheets of a fibrous insulation, e.g., 3/16-in-thick micro-quartz, worked well. A two-in-thick build-up has proved to be sufficient, and a typical installation is shown in Figure 5. This approach can only be used up to about 2000°F main heater temperature in argon and in a vacuum, or 2600°F main heater temperature in air. The 2000°F temperature limit in a nonoxidizing environment is due to the release of siliceous vapors and 2600°F is about the maximum operating temperature for the blanket material.

For higher temperature testing to 3000°F, fine-grain alumina powder (14-20 mesh) was selected as having the required insulating value in a 2-in thickness and this also met the purity requirements. A stainless screen enclosure lined with micro-quartz is used to contain the alumina powder as shown in Figure 6.

2.2 Environmental System

The environmental system consists of a modified vacuum coater, manufactured by NRC Equipment Corporation. The system is equipped with a 24-in-diameter, 30-1/2-in-high, water-cooled, mild-steel bell-jar work chamber placed on a nickel-plated steel base plate. The high-speed vacuum-pumping system, consisting of a 15 CFM Rotary Gas Ballast mechanical pump and a 6-in, 1500-liters-per-second diffusion pump, provided sufficient capacity to evacuate the chamber with a test stack mounted within, 1×10^{-5} mm Hg.

A swivel hoist is provided for lifting the bell jar and allowing it to be swung and lowered to a work bench or storage cart. The hoist also is used to lift heavy stack components (e.g., heat sinks), and may be used to lift an entire assembled test stack. The test stack may therefore be assembled in a remote area and then brought to, and installed in, the test chamber.

Six Conax thermocouple feed-throughs, installed in the base plate, provide for twenty-four thermocouples. In addition there are four sealed water connections, an inert gas or air backfill fitting, four 200-amp power feed-throughs for guard heaters (corners and sides), and six 50 amp power connectors for the main heater and the two auxiliary heaters.

Should a power failure occur during a high-temperature vacuum test, the inert gas backfill line is provided with an automatic solenoid to permit rapid inert backfill, thus avoiding sample oxidation.

A 0-30-in Bourdon dial pressure gauge is provided for use during inert gas backfilling and operation. Between 1 and 10^3 mm Hg., two thermocouple vacuum gauges are provided, one in the chamber and one in the line between the mechanical and diffusion pumps. Below 5×10^{-3} mm Hg., an ionization gauge is used to determine chamber pressure.

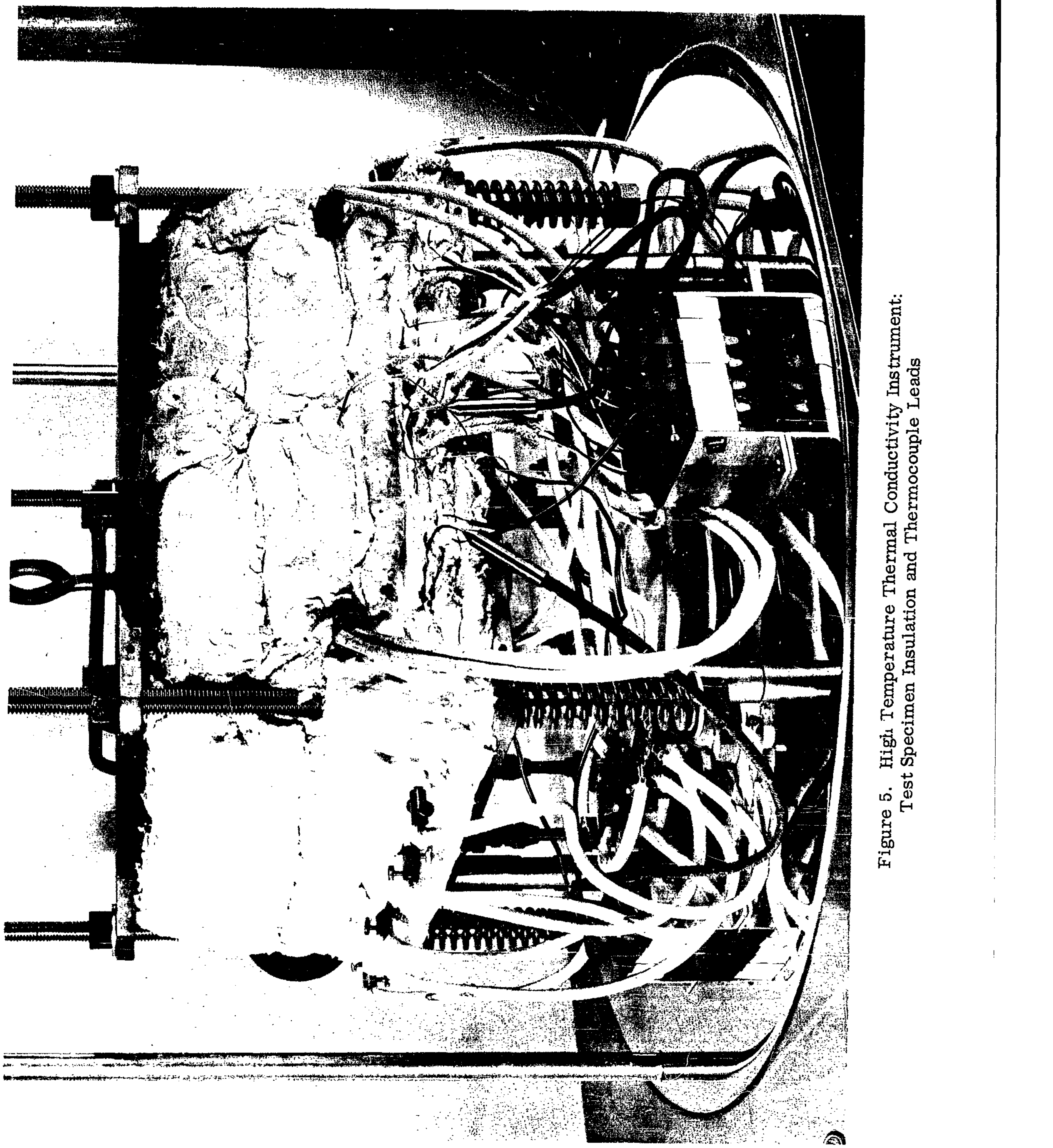


Figure 5. High Temperature Thermal Conductivity Instrument: Test Specimen Insulation and Thermocouple Leads

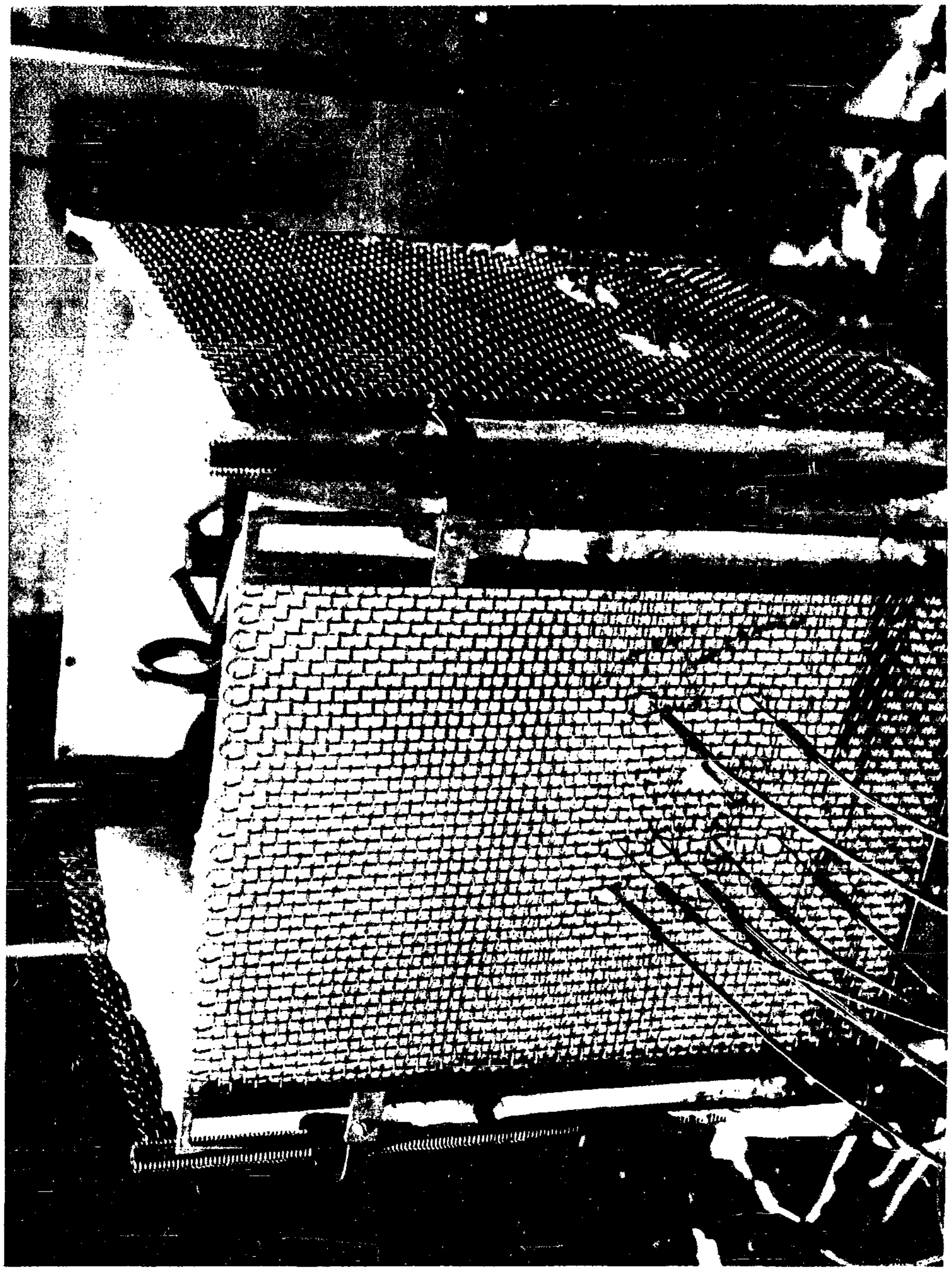


Figure 6. Stainless Screen Enclosure Containing Alumina Powder

2.3 Power, Control, and Instrumentation System

The power and control circuits are shown in Figure 7. Proceeding through the circuit from input, the following components are involved. The main power leads carry approximately 35 KW of three-phase, 220 VAC, to the main fuse box which is followed by the three-phase power supply for the vacuum system and a single-phase 5 KVA auxiliary power transformer (220 to 110 VAC). The auxiliary transformer supplies power for the controllers, the magnetic holding circuit breaker, and any auxiliary power required at the panel.

Next the power lines pass through the main power circuit breaker. This circuit breaker is closed manually and may also be opened manually in case of an emergency. During operation, a magnetic holding coil keeps the breaker closed. The energizing circuit for the main circuit breaker is connected through five over-temperature controllers (two for auxiliary heaters, two for guard heaters, one for main heater), two water-flow interlocks, and a panel switch. The overtemperature controllers indicate the operating temperature of the heaters and are set to open the main circuit when any selected temperature is exceeded. The water-flow interlocks are set to de-energize the main circuit breaker if the water flow to either heat sink falls below4 g.p.m. An override switch allows special stack heating for vacuum off-gassing without the normal water flow. During off-gassing, stack temperatures are monitored manually to ensure safe operation. The panel switch provides a means of breaking the main circuit easily without throwing the master switch. Remote switches may be incorporated in this energizing circuit as desired.

The main power transformer follows the main circuit breaker. This transformer consists of three single-phase, 15 KVA, 220 to 110 VAC, transformers, wired as three-phase open delta.

The five heater circuits connect to the output of the main power transformer. The main heater circuit passes through a 3 KVA constant voltage regulator, an autotransformer for manual control, an ammeter, a wattmeter, and to the main heater.

The two guard heater circuits pass through a solid-state silicon-controlled rectifier, proportional controllers and ammeters to the guard heaters. One circuit feeds the corner guard heaters while the other feeds the side guard heaters. Control potentiometers allow the corner and side guard heaters to be manually balanced with each other.

This independent control of the corner and side guard elements is necessary, since the ratio of edge heat losses from the corners and the sides varies with operating temperature. The control potentiometers provide a means of biasing the power dissipation of the corner and side guard heater relative to each other.

A single proportional controller with null-balance amplifier provides the automatic balance between the main and guard heaters. The sensor for this

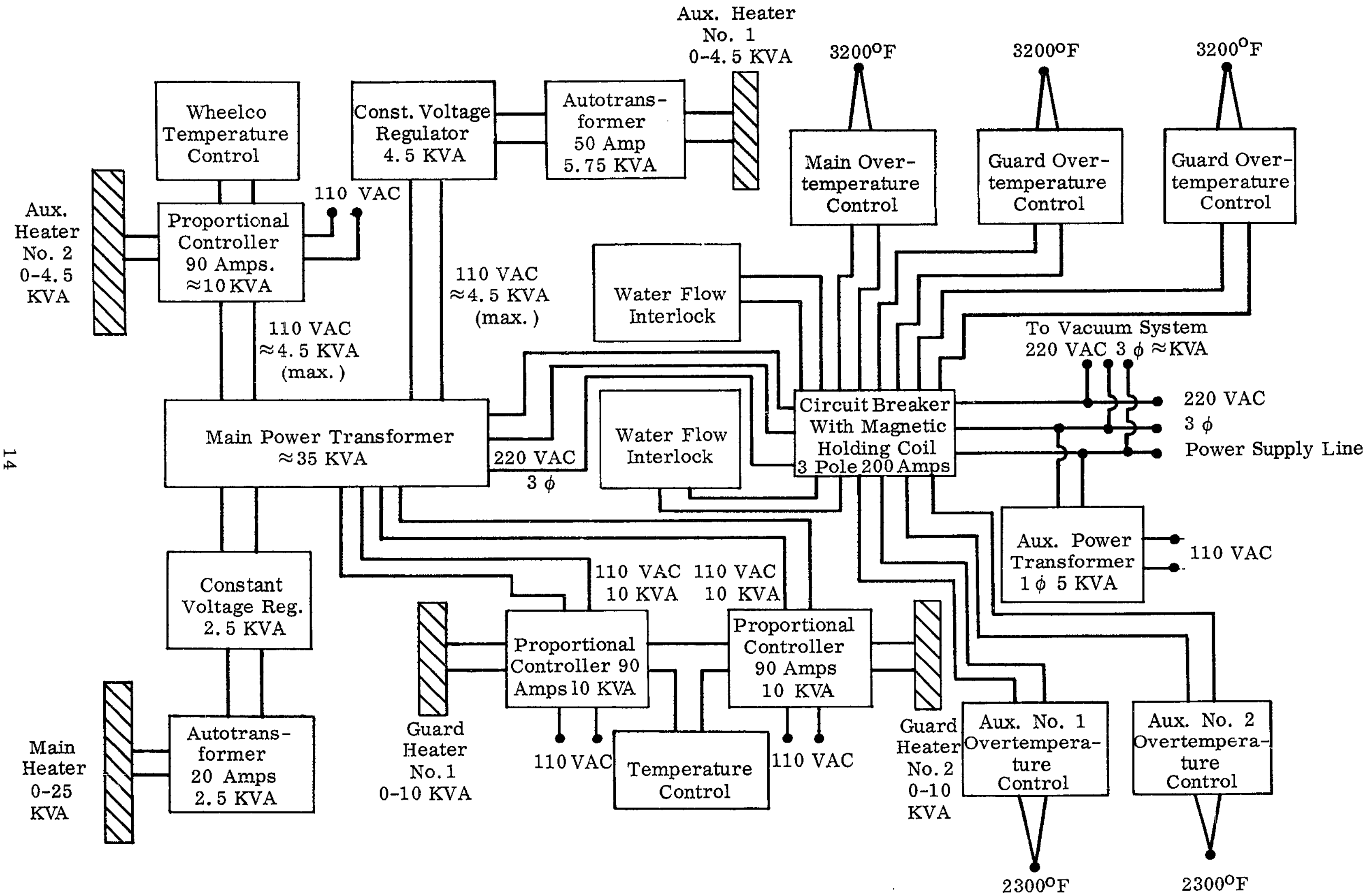


Figure 7. Power and Control Circuits

controller is a 16-junction thermopile placed on the surface of the test sample. The measuring junctions are placed under the main heater while the reference junctions are placed under the guard heaters. A zero signal indicates a balanced condition with all junctions at the same temperature, on the average. The use of a thermopile or star-multiple thermocouples for maintaining balance of a thermal conductivity apparatus of this type is common practice. The controller visually indicates the degree of balance.

The circuit to auxiliary heater No. 1 is identical with the main heater circuit with the exception of a greater current-carrying capacity and the replacement of the wattmeter with an ammeter. Auxiliary heater No. 2 is automatically controlled by means of a proportional controller. The control sensor for this circuit consists of four thermocouples mounted on the surfaces of the two ratio elements. The outputs of the two thermocouples on a single ratio element are added; this signal is then subtracted from the sum of the outputs from the two thermocouples mounted on the other ratio element. Equal average temperatures for both ratio elements result in a null condition. Any unbalance is indicated by a net signal, its polarity indicating the direction of unbalance.

The primary instrumentation consists of a precision thermocouple potentiometer and a single-phase wattmeter. All temperatures and temperature differences which appear directly in the reduction of conductance data are obtained manually with this potentiometer. The heater overtemperature controllers and null-balancers serve only to provide a rough visual indication of operating conditions. The power to the main heater is read directly on the wattmeter. The ammeters in each circuit serve only to indicate rough power settings to aid the operator in reaching test conditions.

All of the controls and power equipment are contained in steel cabinets. The wattmeter and potentiometer are mounted in a desk panel which also provides a writing area for the operator.

Section 3

TEST PROGRAM

A total of eight tests have been completed during the testing and calibration phase of this program. Two calibration tests, one on copper and the other on alumina have been conducted twice, first in Dynatech's laboratory and then again at Wright-Patterson Air Force Base after the apparatus was installed. Calibration tests on two refractory metals remain to be completed.

The following table presents a summary of the tests performed, including a description of the test conditions and the temperature ranges.

Table I

Material	Environment	Temperature Range ^{o}F	Remarks
1. Copper	Argon	600 to 1800	Run Twice (Calibration Test)
2. Aluminum Oxide Al_2O_3	Air	925 to 3025	Run Twice (Calibration Test)
3. René 41 Honeycomb	Air Vacuum	780 to 1640 1080 to 1525	--- 5×10^{-5} mmHg
4. Titanium Honeycomb	Air Vacuum	325 to 905 530 to 1000	--- 5×10^{-5} mmHg
5. L-605 Honeycomb	Air Vacuum	610 to 2100 960 to 1950	--- 5×10^{-5} mmHg
6. Precipitation Hardening Stainless Steel	Air Vacuum	395 to 1020 960 to 980	--- 3×10^{-4} mmHg
7. René 41	Air Vacuum	390 to 1840 975 to 1575	--- 1×10^{-4} mmHg
8. Fused Silica	Air Vacuum	430 to 2550 840 to 2000	--- 2×10^{-5} mmHg
9. Tungsten and Columbium	Argon	Not Completed	

All of the data obtained during these eight tests fall well within the specified accuracy when compared with generally accepted reference data.

Two rather serious problems have been encountered during the test program. The first concerned the reliability of the main and guard heater assembly. The second problem involved the installation of surface thermocouples in the test samples. Because of the important nature of these two problems, they are discussed in detail in the following two subsections.

3.1 Heater Development

The specifications set forth for the thermal conductivity instrument as described in this report called for the design, development, fabrication, and delivery of a special high temperature heating element which, while not new in concept, was certainly unique in application. This heater element was to have met the requirements necessary to satisfy the instrument specifications. These included a maximum temperature of 3000^{o}F, a maximum heat flux of 40 watts/in^2, and operational environments of air, inert gas, or 10^{-5} mm Hg.

The original concept for the high temperature heater as proposed by Lexington Laboratories, Inc., as a subcontractor, was to construct an electrical resistance heater having a geometry similar to that shown in Figure 1, and consisting of only two materials of construction, pure aluminum oxide and an alloy of platinum versus platinum-40%-rhodium. The heater blocks were to be machine grooved from solid 4 in x 4 in x 3/4 in. alumina blocks. Into these grooves would be wound 0.041 in. diameter heater wire and a slip casting of pure alumina applied over the wire to fill the grooves completely.

In an attempt to prove the feasibility of this heater design, two model heater configurations were constructed by Lexington Laboratories. The first model was a 1 in x 1 in scaled-down heater in which the proposed heater materials would be used and the fabrication techniques would be developed. A total of five of these model heaters were constructed. These models were subjected to tests which were to have been more stringent than actual conditions for the final heater. During these tests, heaters were operated at temperatures in excess of 3200^{o}F, operated in vacuum and air, operated at 110% of rated power, and operated for continuous periods of approximately 1,000 hours. In only one instance did a model heater fail. Vibration, caused by a vacuum pumping system and the induced fatigue, caused one heater lead to fail. Several heaters were broken open after varying periods of operation. A small amount of evaporation and metallic crystal condensation was noted around the heater windings after approximately 100 hours. This apparently reached an equilibrium condition since heaters that operated several hundred hours appeared no worse than those which were opened after 100 hours. No other detrimental effects were noted during these small heater tests.

A second, lower temperature, model heater was constructed by Lexington Laboratories to check the over-all heater configuration. This second heater was a full scale heater in which the alumina blocks were replaced with fired lava, the platinum-rhodium wire was replaced by KANTHAL wire, and the alumina slip was replaced by an Alundum cement. It was planned that this heater was

to be used in a model stack configuration to check out the actual heater configuration and the thermal guarding concept. Due to a delay in the delivery of this heater, the actual thermal conductivity instrument and test stack were available at the time this model heater was delivered. In order not to delay the test program, and since the final heater was not available, it was decided that the copper calibration test would be conducted using this lava KANTHAL heater. During this unsuccessful test, which was conducted in an inert (argon) atmosphere, every heater element became discontinuous, causing heater failure. Upon examination, it appeared that a severe attack on the heater wire occurred, probably originating from an interaction of the trace materials present in the cement in the absence of oxygen (air).

An alumina, platinum-rhodium heater, as previously described, was delivered by Lexington Laboratories. This heater element was used with fair success during the copper, René 41, stainless steel, alumina, fused silica, and honeycomb tests described in more detail in the following sections. Several failures did occur during these tests, however. Numerous lead failures appeared, usually during cool-down.

The exposed platinum-rhodium lead wire became quite brittle after a short period at high temperatures. This was explained partially by grain growth and partially by silicon attack. The silicon originated from the breakdown of trace siliceous materials present in high temperature regions during vacuum or inert gas operation. Although a conscientious attempt was made to keep all siliceous materials away from hot regions, since the silicon-platinum reaction is well-known, only partial success was realized because almost all ceramics have traces of silicon present.

The second failure mechanism was a sudden discontinuity of heater elements, also usually occurring during cool-down. Location and examination of the break indicated a mechanical tension failure of the wire within a groove and a parting of the two ends by a several-thousandths-inch gap. Alumina and the platinum-rhodium alloy have fairly close coefficients of expansion, and the method of imbedding the wire should have prevented mechanical stress due to differential expansion.

In some instances cracks appeared in heater blocks. These cracks appeared both parallel and normal to the grooves. These cracks may be partially blamed on the method of separating the heater from the sample during several of the tests. The heater blocks were supported at the four corners by small ceramic spacers placed between the blocks and the samples. Some heater warping was also observed as a result of this support method. Although cracking did not completely disappear when the heater was fully supported, it was substantially reduced.

Because of the general difficulty with the Lexington Laboratories' heater, particularly in regard to the heater lead problem, it was decided that Dynatech would undertake a complete redesign and the necessary research and development required to provide a heater, or heaters, compatible with the requirements of the instrument specifications. The test program, with the exception of the columbium and tungsten tests, was complete at this point.

The heater assembly was completely redesigned, this time using pure alumina blocks machined in the green state and then fired, thus avoiding the extremely expensive machining cost required in the other design. Furthermore, holes were provided through the center of the blocks into which lead supporting alumina tubes were placed. The heater lead problem was completely eliminated with this new design.

The new heater element was fabricated and an attempt was made to test the columbium and tungsten samples. This test is described in Section 3.11. Several heater blocks failed during this test at approximately 2000°F and the blame was placed on porous slip and columbium contamination. At the time this heater was constructed, a good grade of alumina slip was not commercially available, nor was the slip used in the earlier heater. An examination of the heater wire showed evidence of contamination blamed on the columbium and the poor protection due to the high porosity of the alumina slip. This was further substantiated by the very strong reaction between platinum-rhodium sheathed thermocouples installed directly in the columbium blocks.

The continuous blocks were salvaged and replacement blocks constructed, this time using a very good dense slip. It was felt that this new dense slip would afford sufficient protection for the heater windings.

A second attempt to run the columbium-tungsten test met with a similar failure of all heater blocks at approximately 2300°F. A thorough analysis of the heater windings indicated the following. The heater windings had failed in two distinct manners, and each heater in many places: first, breaks having the appearance of pure mechanical failures as previously described, and second, breaks in which there was strong evidence of contamination. In the latter, the heater winding tapered to points on either side of the break and the wire surface had a very rough texture, as in almost always the case when contamination has taken place.

In a parallel program, undertaken by Dynatech to measure the thermal conductivity of graphite in an apparatus very similar to this instrument, a heater failure, similar to the failure which occurred during the columbium and tungsten tests, was encountered. In this case the failure occurred in the same manner as that described above. In still another instrument, a heater with a different geometry (cylindrical) but using the same materials has been operated to over 3000°F in vacuum and in the presence of both carbon and silicon vapors. This heater has operated almost continuously for a six-month period without a single failure. This instrument, however, operates at a much lower heater winding power density and is on manual control. The cycling of the proportional guard heater controllers might thus contribute somewhat to the failures.

Currently, Dynatech is continuing further research with the goal of developing a heater, or heaters, which will meet the requirements for this instrument. It presently appears that the platinum-rhodium heater is acceptable for air operation under all specified conditions, and vacuum or inert gas operation to approximately 2200°F at high heat fluxes and 3000°F at very low heat fluxes.

Tests are now being conducted at Dynatech on heaters using refractory metals for use in vacuum or inert gas. Three tests have already been conducted with a molybdenum-wound heater to $2700^{o}F$ in the presence of graphite. A lead failure occurred during the third test. A tantalum heater is presently being constructed and will be subjected to similar tests. If contamination problems still exist, a feasible heater may be developed for reasonable life in which the heater winding is exposed, thus permitting occasional rewinding of the heater blocks.

Several apparent anomalies appeared which to date have not entirely been clarified; however, it presently appears that several factors contribute to failure of heater elements. None of these factors alone cause heater failure. The following contribute to failure:

1. Temperature Level
2. Power Level (heat flux)
3. Environment
4. Contamination

To illustrate the apparent anomaly consider the following:

1. <u>Temperature Level</u>

   During the alumina test (in air) the heater operated satisfactorily on two occasions to over $3000^{o}F$.

2. <u>Power Level</u>

   During the copper calibration test the heater operated twice (in inert gas) satisfactorily at 20 W/in^2 heat flux to over $2000^{o}F$. Tests on small model elements ran satisfactorily at 110% of rated power, for 1,000 hours at temperatures in excess of $3000^{o}F$.

3. <u>Environment</u>

   Successful tests have been conducted in air, inert gas (argon) and vacuum (approximately 1×10^{-5}mm Hg).

4. <u>Contamination</u>

   The fused silica test was successful to approximately $2600^{o}F$ in air and vacuum and attempts to measure columbium did not affect the heater until the heater was operating above $2000^{o}F$. Furthermore, Dynatech has operated an apparatus with a cylindrical heater to over $3000^{o}F$ in carbon and silicon atmospheres and this apparatus has operated almost continuously for six months.

Two attempts have been made to determine the thermal conductivity of columbium. Each attempt has been met with a heater failure. In addition, an attempt to measure graphite in a similar Dynatech instrument has met with the same failure under identical conditions. It presently appears that each of the four factors listed above contribute to the heater failure. In both columbium tests and in the graphite test, the failure occurred at between 2300°F and 2600°F, whereas the heater performed well under other conditions to 3200°F. The heater in each failure was delivering approximately half of its designed heat flux, although other tests on small model elements had operated for almost 1,000 hours at 110% of rated power at over 3000°F. Furthermore, the copper calibration test required the same power density at 2000°F that later caused failure at a higher temperature.

Tests run on fused silica caused no problem in air or vacuum at temperatures above 2500°F, although silica is well known to break down in vacuum at these temperatures and has a strong ability to contaminate platinum alloys. (Silicon attack was partially to blame for some of the early Lexington Laboratories' heater lead failures.) The silica test was run at a very low heat flux (power density) however.

Other experiments at Dynatech have shown that carbon contamination in vacuum has no major effect on platinum-rhodium at 3000°F, provided the heater operates at a much lower heat flux (power density), but failures occur at around 2500°F when heaters operate at high power densities.

To summarize, the results of the various tests with this instrument and other Dynatech-sponsored research indicate that the Lexington Laboratories' heater concept will meet only the following test conditions:

1. <u>Air Operation</u>

   Maximum heat flux: 20,000 Btu/hr-ft^2
   Maximum temperature: 3000°F

2. <u>Vacuum and Inert Gas</u>

   Maximum heat flux: 5000 Btu/hr-ft^2
   Maximum temperature: 3000°F

3. <u>Vacuum and Inert Gas</u>

   Maximum heat flux: 20,000 Btu/hr-ft^2
   Maximum temperature: 2000°F

These conditions appear allowable even with materials which react with platinum-rhodium. The exact degree or extent of the effect of contamination is not known. It might be that the above conditions may be relaxed for some test materials or are not conservative enough for others.

3.2 Sample Instrumentation

Early in the test program, it became apparent that special thermocouple fabrication and installation techniques would have to be developed to meet

the temperature measurement requirements of this program. There are no "standard" thermocouple installation techniques that would be satisfactory. The necessary care with which the sample surface temperatures are measured stems from the fact that the accuracy of the determined conductivity values is directly proportional to the accuracy with which the temperature drop across the sample is determined.

Installed in the apparatus are provisions for either chromel-versus-alumel or platinum-versus-platinum -rhodium thermocouples. Both calibrations have been used during this program.

The problems associated with the thermocouples may be broken into two categories. First, a suitable thermocouple must be selected. Second, a suitable method of installation must be established. The first problem stems from the fact that the thermocouple must remain intact, providing an accurate signal corresponding to the temperature at its junction throughout the test. Thermocouple drift due to contamination and discontinuity due to wire breakage must be avoided. The second problem arises from the fact that the thermocouple junction must be equivalent in temperature to the selected point of measurement on the sample, usually the surface.

Many thermocouple characteristics not found in the general literature have been determined during this program, in most cases by trial and error.

To summarize the general limitations of the various types of thermocouples as encountered during this program, the following conditions are listed:

1. Unsheathed (bare) chromel-alumel thermocouples may be used to temperatures of 2000°F in air and 1500°F in vacuum or inert gas.

2. Unsheathed platinum-platinum-rhodium thermocouples may be used to 3000°F in air and to 1500°F in vacuum and inert gas.

3. Sheathed chromel-alumel thermocouples may be used to 2000°F in vacuum and inert gas provided the metal sheath is not attacked by surrounding materials.

4. Platinum-sheathed, platinum-platinum-rhodium thermocouples may be used to 3000°F in vacuum provided the platinum sheath is not attacked by the material in which the sheath is imbedded.

The above vacuum limitations, imposed in 1 and 2, apparently are due to attack by materials breaking down at these temperatures in vacuum. Although it is not certain, the worst contamination problem appears to be due to the breakdown of siliceous materials and a silicon-platinum reaction. The result is a gradual drift in thermocouple output and an embrittlement often causing a failure during cooldown. Another series of tests in an unrelated program conducted in our laboratory indicates that under vacuum or inert gas, carbon vapor originating from

graphite samples causes the same observed contamination. With carbon vapors present, platinum not only is reacted on by the carbon but also an unexplained platinum-alumina reaction has been observed.

The problem of thermocouple installation has also proved to be a considerable one. Altogether five thermocouple installation techniques have been required to perform the tests described. These techniques are as follows:

1. With high conductivity materials, tested in the absence of thermocouple contamination (in air, or in vacuum below 1500°F with chromel-alumel and 2400°F with platinum-rhodium), the optimum installation appears to be accomplished by grooving the sample to receive a 1/8 in. diameter pure alumina, two-hole ceramic tube lying flush below the surface. The junction is peened into the surface of the test sample. This technique was used in the René 41 and the Precipitation Hardening Stainless Steel test. This method was also used to install the star differential thermocouple to control the guard heater during the copper test.

2. With high conductivity materials, tested in vacuum or inert gas to temperatures in excess of 1800°F with chromel-alumel and 3000°F with platinum-rhodium, commercial sheathed thermocouples were used successfully. These were installed and cemented in grooves similar to the first method of installation. This method was used during the copper test and the alumina test.

3. With low conductivity materials that are not electrically conducting, it was found sufficient to merely lay bare thermocouple wire on the test specimen, cementing the junction to the sample. This technique was used in the fused silica test. The technique was also used to install the differential star thermocouple on the honeycomb samples after coating that portion of the sample under the wire with an electrically insulating cement.

4. During the first columbium test, platinum-sheathed, platinum-rhodium thermocouples were installed in 1/8 in. grooves as described in (2.) above. During this test, it was found that there was a strong interaction between the platinum sheath, its contents, and the columbium surrounding the sheath. It appeared that the platinum had formed a low melting point eutectic with the columbium. During the second columbium test, bare platinum-rhodium thermocouples (0.020 in wire) were placed in continuous, 1/8 in, one-hole, pure alumina tubes which lay in 1/8 in surface grooves passing entirely through the test stack into the cold region at opposite sides. These thermocouples showed no apparent attack to temperatures of approximately 2500°F. The temperature values obtained from these thermocouples, however, indicated a temperature difference across the sample that was approximately 50% greater than anticipated. A subsequent test in our laboratory using an aluminum block explained this discrepancy. With the

extremely high heat fluxes used to establish a reasonable temperature difference across a reasonable thickness of high-conductivity material, large isotherm distortions are experienced around the relatively low-conductivity, alumina insulating tube located at the boundary of the sample. Potential theory predicts that potential lines are distorted for a distance of approximately 1.1 diameters in the flow direction from a cylinder located in a streamline flow. An analysis showed that if the ceramic insulators were located at least 1.1 tube diameter below the sample surface, the measured temperature would be correct. Four thermocouples were installed in an aluminum block, two in tubes at the opposite surfaces and two in tubes located 1.1 diameters below the surfaces. The aluminum block was tested in a comparative thermal conductivity apparatus in our laboratory. As predicted, the surface thermocouples indicated an error of approximately 50% in the measured thermal conductivity, whereas the submerged thermocouples determined a thermal conductivity value for aluminum within 5% of reference values.

5. The honeycomb samples posed a rather difficult instrumentation problem. The structural surfaces or face plates of these honeycombs ranged in thickness from 0.025 in to 0.050 in. It therefore was impossible to groove these surfaces to receive thermocouples. Thus, in order to provide space for the thermocouples to pass into the stack within ceramic tubes, the heater elements were separated from the sample by small 1/4 in x 1/4 in x 1/8 in. alumina pads located at the four corners of each heater element. Similar pads were used to separate the samples from the ratio elements. Heat transfer to and from the sample was accomplished primarily by radiation.

   Good thermal contact with the sample is usually done by peening the thermocouple junction into the surface. In view of the thin face plates, this was impossible with the honeycomb structures. The general technique in installing thermocouples is to not only ensure that the thermocouple makes good thermal contact with the surface being measured, but also to bring the thermocouple leads out in such a manner that they do not go through a temperature gradient in the immediate vicinity of the junction. If a gradient is imposed in the lead wire near the junction, a considerable error can be introduced. A preliminary test with the stainless steel samples produced a conductivity error of 20 to 30% when bare wire thermocouple junctions were peened into the surface and the wire brought up immediately out of the block and into an insulator. Merely by bringing the thermocouple insulator down into a groove in the sample, thus eliminating the gradient, accurate measurements were produced.

   With the honeycomb samples, shallow slots were milled in the surfaces 0.020 in. deep, 1/16 in. wide and about 1/2 in. long.

The thermocouples were then installed as shown in Figure 8a. Before filling the slot with ceramic cement, the junction was pushed up against the bottom and side of the slot, and its metallic contact checked with an ohmmeter. A small sliver of mica was inserted into the groove between the wire and the sample to prevent contact at any point except the junction.

In the first honeycomb test three thermocouples were installed on each surface to check for surface temperature variations due to the inhomogenity of the structure or honeycomb below the face plate. The thermocouple readings varied less than 1/2 %; therefore, only a single thermocouple was used in subsequent tests.

3.3 Copper Calibration Test

The required accuracy of the device at 1800°F was specified as ± 5%, and in order to show such a degree of precision, the thermal conductivity of copper has been measured at several temperatures in the range between 400° and 1800°F. As a criterion for the accuracy, the conductivity-versus-temperature curve as published in WADC TR 58-476 (Reference 2) is considered to be the most representative. The sample was 2-1/2 in. thick, 99.95% pure, electrolytic, tough pitch copper. In this test only one sample was used in a stack as shown in Figure 4 of Section 2.1.2. Due to the high conductivity of copper, the temperature drop across the sample is very small even at maximum heat flux. Inaccuracies in the measurement of the surface temperatures and the limited degree of precision with which a potentiometer can be read, bear a great influence on the quality of the final answer. The temperature drop across the copper was measured with two chromel-alumel thermocouples, each contained in a 1/16 in. stainless steel sheath. The couples are permanently cemented in grooves that have been machined parallel to the surface on each side of the sample. The test was run in an environment of argon at atmospheric pressure. Figure 9 shows a top view of the installed samples.

The results, as shown in Figure 10 indicate an accuracy well within the prescribed ±5% limits over the entire temperature range, where measurements were taken. This test was run first in Dynatech's laboratory and then again after installation at ASD.

3.4 Alumina Calibration Test

The alumina test was conducted with two test samples, 5/8 in. thick. Conductivity measurements were performed, however, on one sample only, while the other was used to control the input to the guard heaters. At the highest temperature these duties were switched and several points have been obtained for the latter sample as well, (the points enclosed by a triangle in Figure 11). As expected with two identical samples of a dense material, the results from both measurements coincide very closely. The entire test from the lowest test point at 900°F to the highest point at slightly over 3000°F was conducted in one run, merely by changing the power input to the different heaters. This demonstrates clearly the tremendous advantage in the use of auxiliary heaters. Time and effort

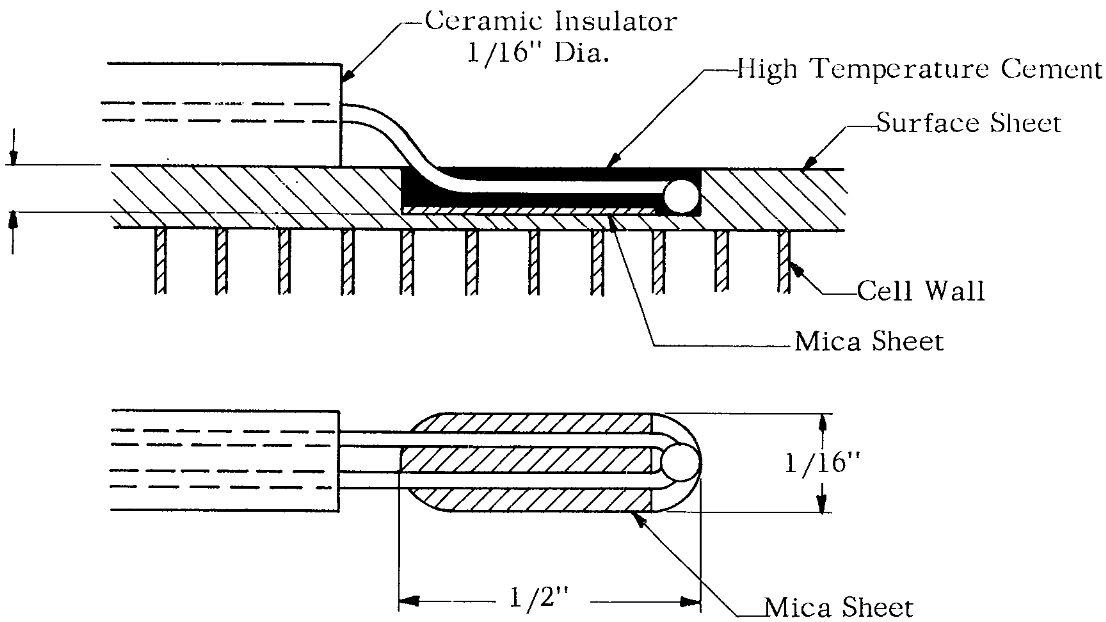


Figure 8a. Schematic Showing Method of Thermocouple Installation in Honeycomb Surface Sheet

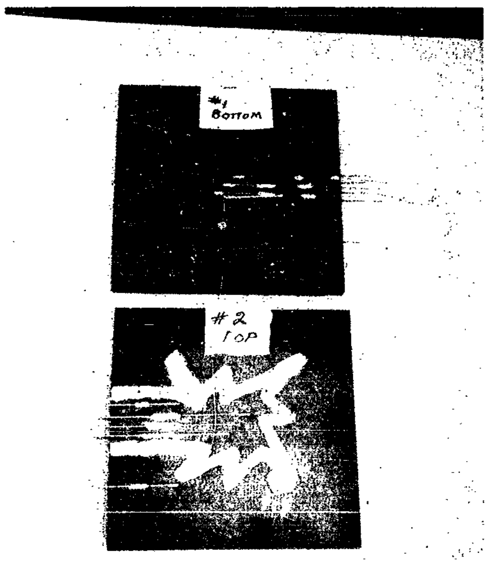


Figure 8b. Thermocouples Attached to the Surface Sheet of a Honeycomb Test Sample

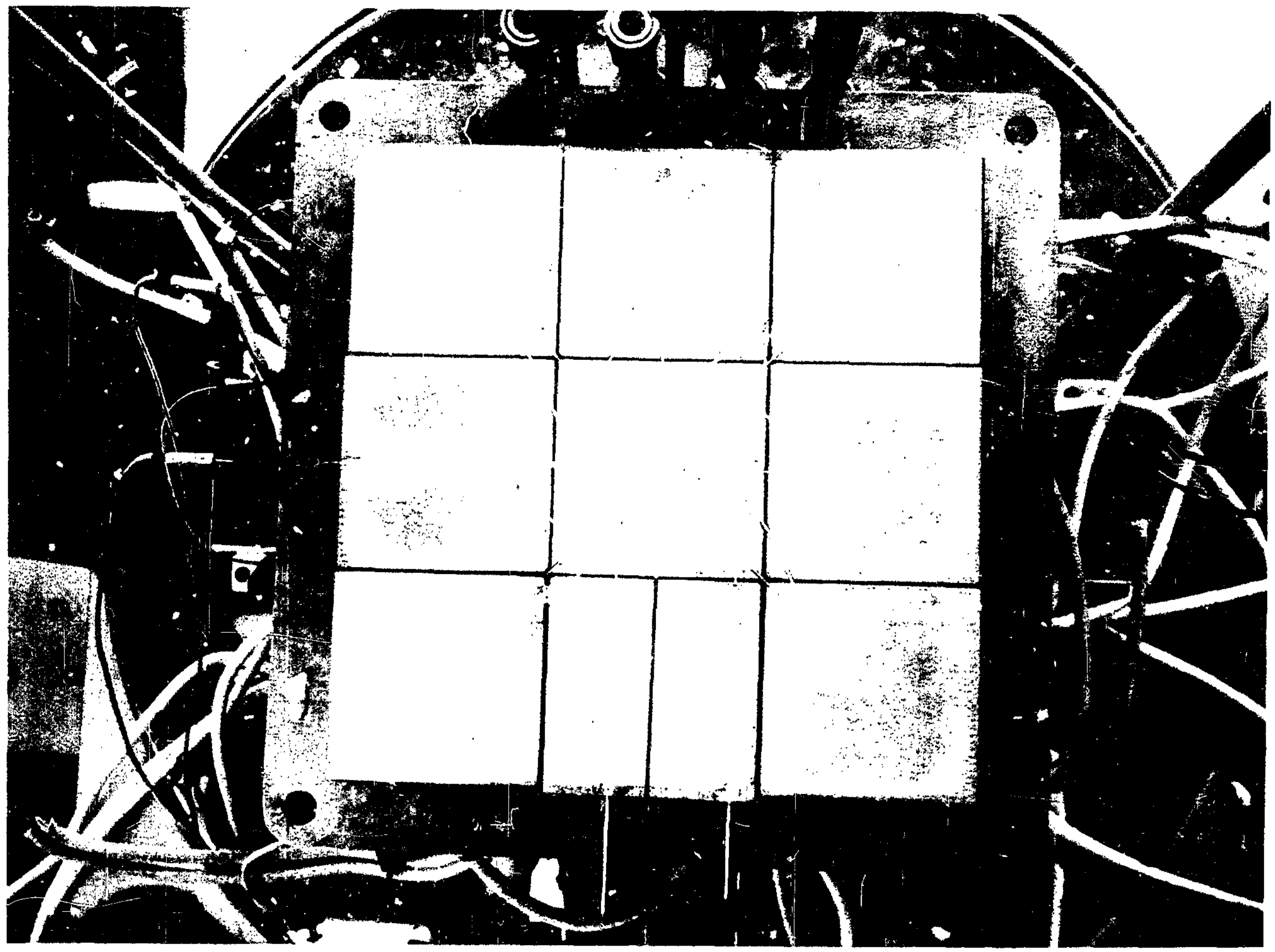


Figure 9. Instrumentation of Solid Copper Sample (Note Thermocouples Below Surface)

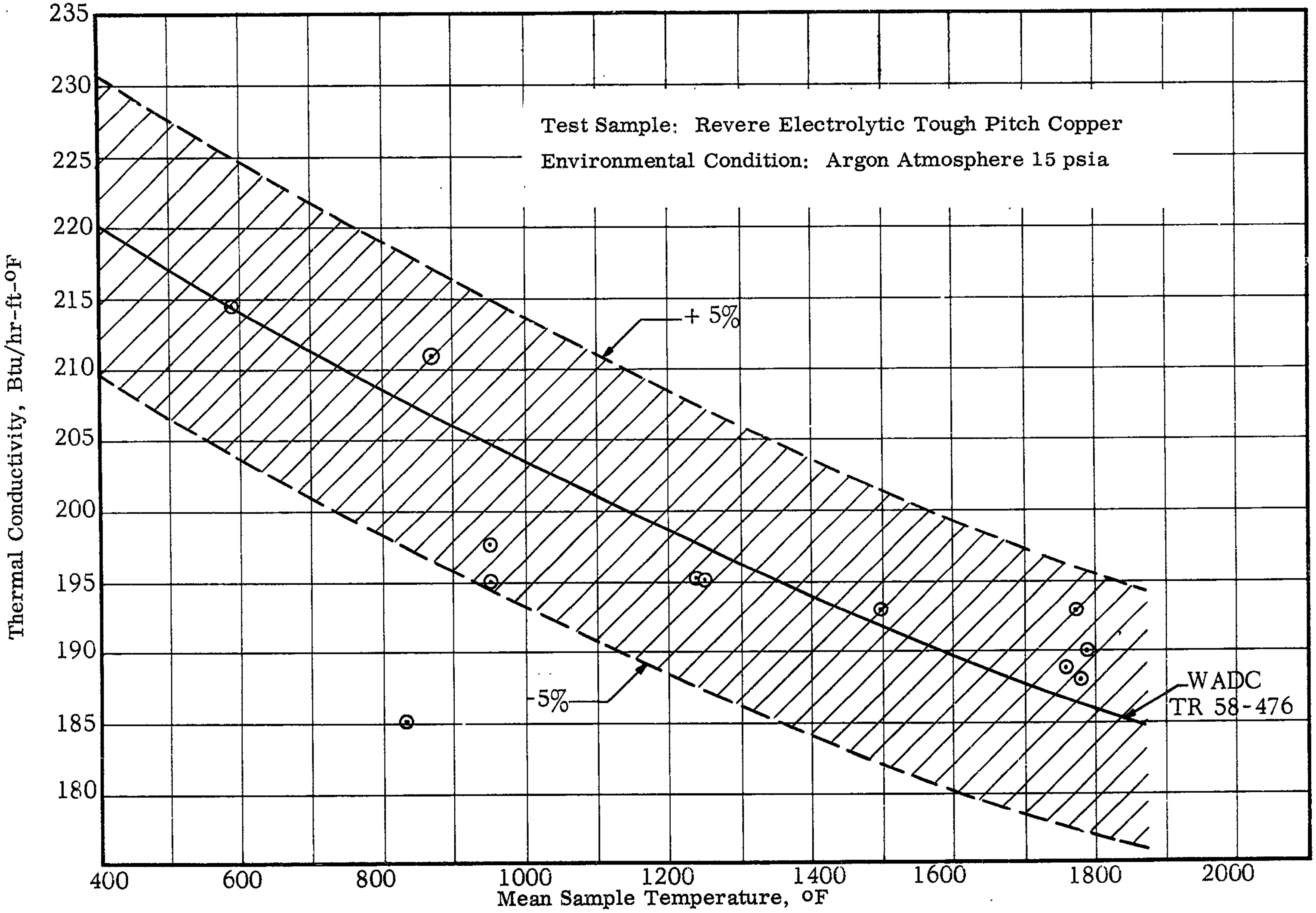


Figure 10. Copper Calibration Test Results

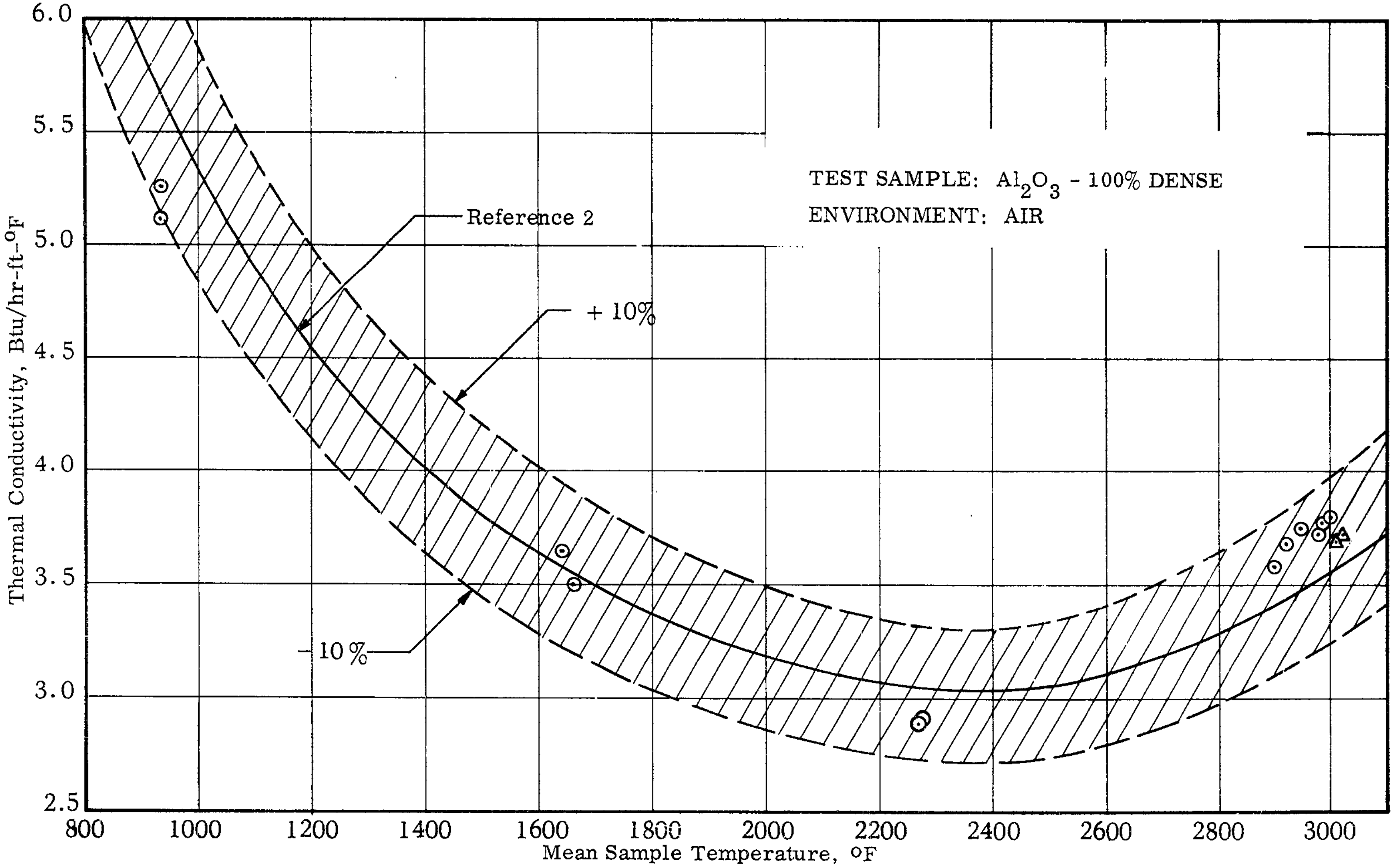


Figure 11. Alumina Calibration Test Results

are saved by not having to take the stack apart half-way in the test range to change the amount of insulation between the sample and the heat sink.

The results are shown in Figure 11, where the data are compared with the curve for dense aluminum oxide as published in WADC TR 58-476 (Reference 3). Each test sample was instrumented with four platinum-versus-platinum-13%-rhodium thermocouples, sealed in a 1/16 in. diameter platinum sheath. Two were in the center block, one on each surface and a couple was attached to the hot sides of one corner block and one side block. Although it would have been permissible to use a star differential thermocouple with 16 junctions out of, e.g., .020 in. diameter platinum vs. platinum-13%-rhodium bare wire, since the test ran in an air atmosphere, it was decided to use an alternative method. The hot side center couple on one of the samples, in conjunction with either the corner or side thermocouple, created a differential thermocouple whose signal could be used to control the guard heater input. The guard side and corner heaters were balanced as usual, after determination of the hot face temperatures of a side and a corner block of the second sample (on which also the temperature drop was measured). Figure 12 shows pictures of the alumina test stack, surrounded by powdered insulation.

3.5 René 41 Honeycomb Test

The over-all thermal conductance of a René 41 honeycomb sandwich panel has been measured at various temperature levels up to 1600°F. Two sets of data have been obtained, one in air and one in a vacuum environment. In air, the transfer of heat from one surface of the panel to the other occurs by means of conduction through the metal core, radiation between metallic parts and conduction and natural convection through the air in the cells. It would be expected that the natural convection increase the conductance when heat is transmitted from the bottom of a horizontal sample to the top and cause a decrease in conductance when the heat path is reversed. The conductance measured in a vacuum environment is therefore independent of the orientation of the honeycomb panel with respect to the heat source.

The two test panels were 12 in x 12 in square with an over-all thickness of .510 in. The two surface sheets were .025 in. thick and the core consisted of square cells with 1/4 in. cell size made out of .002 in. thick foil.

The controlling signal for the guard heater output was obtained from a .004 in. diameter platinum-versus-platinum-13%-rhodium star-thermocouple attached to the hot side of one of the samples. The star-thermopile could not be in electrical contact with the specimen; therefore, a thin coating of insulating cement was applied locally to the surface. The junctions of the star were also cemented to the surface. The surfaces of the honeycomb were sandblasted very slightly to roughen the surfaces sufficiently so that the cement would stick.

Figure 13 shows pictures taken during different stages of the stack build-up.

The effective thermal conductance is calculated from $h = \frac{Q/A}{\Delta T}$, where the temperature drop is computed as the difference between the mean temperatures of each surface. As mentioned before, the effective thermal conductivity

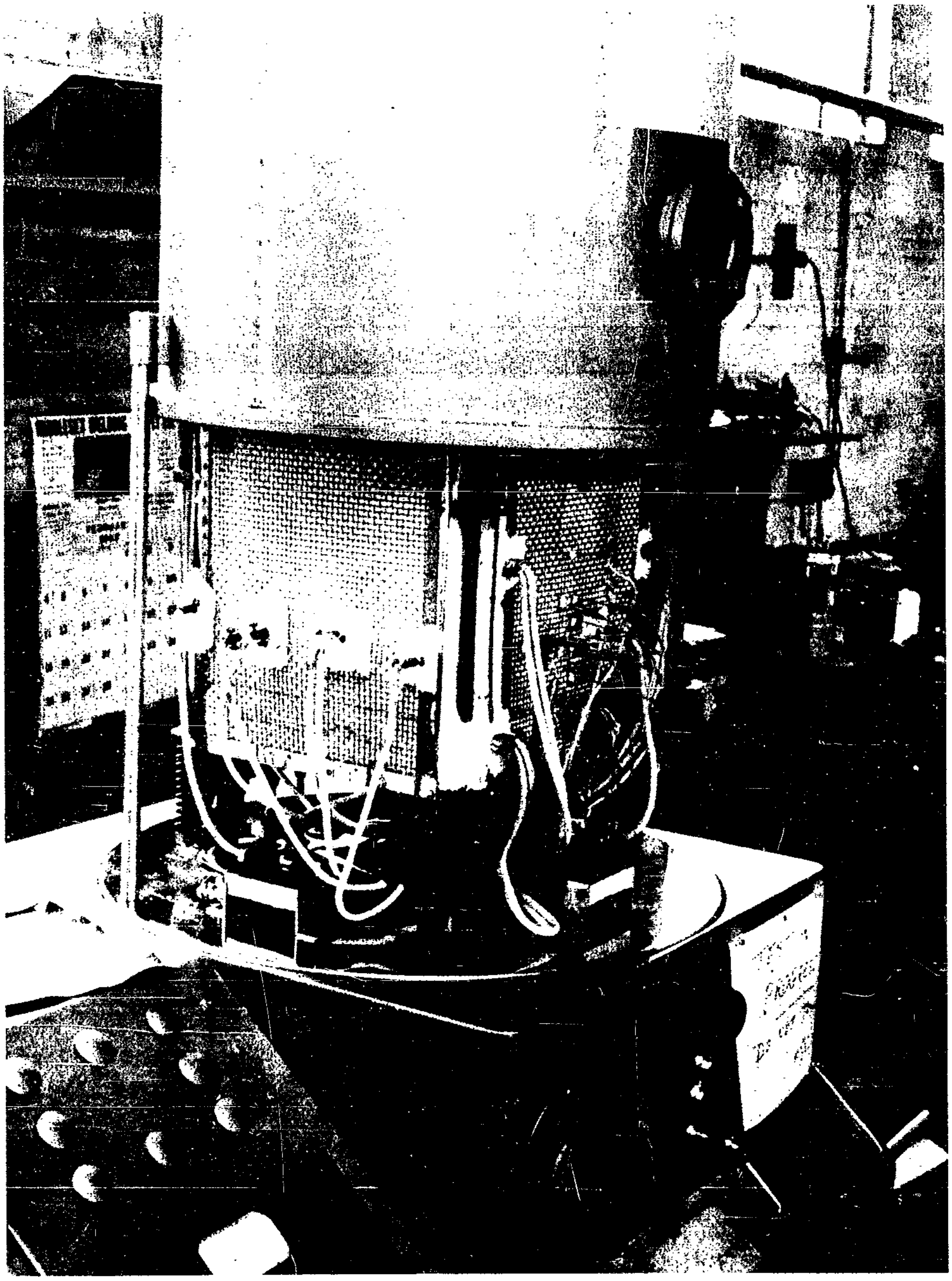
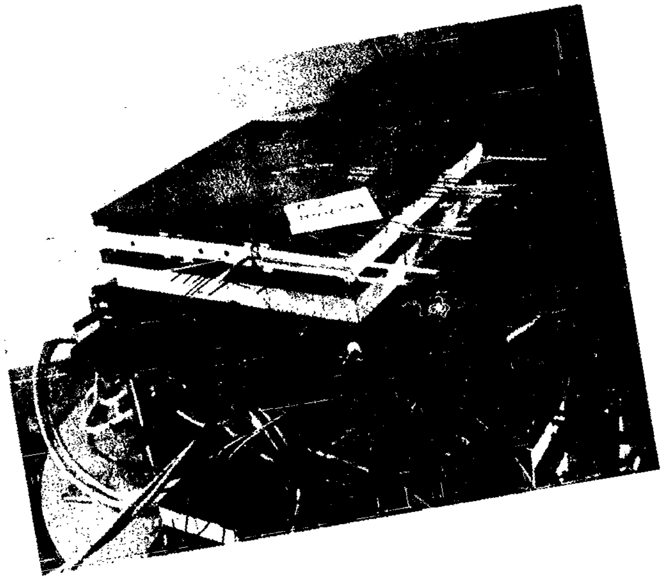


Figure 12. Alumina Test Stack Surrounded with Powdered Insulation



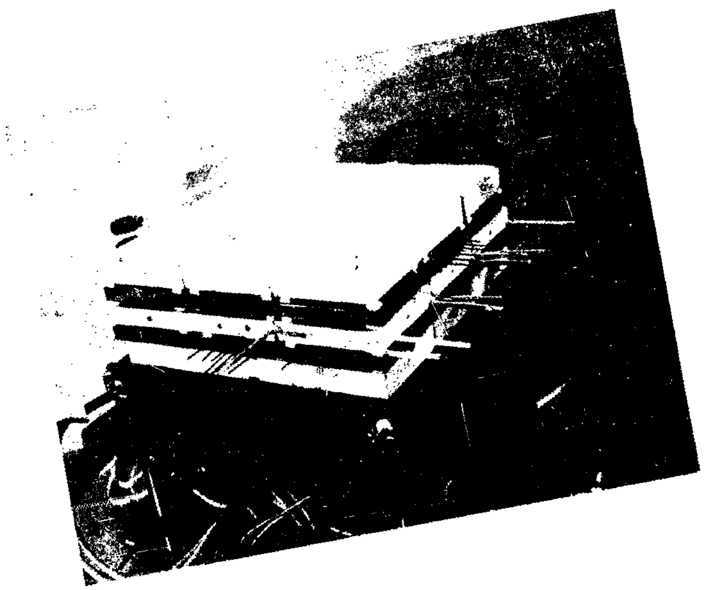


Figure 13. Pictures Taken During Different Stages of the Stack Build-Up

consists of three terms; a radiation term, a term relating the conduction through the core material and a term to account for the heat transferred by the air inside the cells. Especially at the higher temperatures the transfer by radiation becomes predominant. Since the heat transferred by radiation is proportional to the difference between the fourth power of the face temperatures, it can be shown that the conductance pertaining to a particular average panel temperature may vary, depending on the magnitude of the temperature drop even though the average temperature is constant. During this test, efforts were made to keep the temperature drop small in order to approach the limiting value for the conductance.

Values for the conductance are plotted in Figure 14. There is a consistent tendency for the points belonging to the bottom sample to be slightly below those for the top sample, thus verifying the assumption of the effects caused by the natural convection of the air inside the cells. Also it is noticed that the conductances measured in a vacuum are the same for both honeycombs within the accuracy limits of the apparatus. However, many more data points would have to be obtained, in air as well as in a vacuum, before one would be justified in separating the data into different curves.

Also plotted in Figure 14 are the results of calculated effective thermal conductances. At various temperatures the conductance has been calculated according to the method given by R. T. Swann (NASA TN D-171, Reference 4), using the same face temperatures as the ones measured during the test at these temperature levels. The method of Swann does not take into account the effect on the heat transfer due to natural convection.

For small temperature drops, the heat transferred by radiation may be considered proportional to $T_{avg}^3 \Delta T$, and the heat conducted through the core is proportional to essentially a constant times ΔT, if the conductivity of the material varies only slightly with temperature. Therefore, in a vacuum it might be expected that the conductance of a honeycomb panel be equal to a constant plus a constant times T_{avg}^3. The dependence of the effective conductivity on the temperature based on this assumption is also shown in Figure 14. The line is intended only for comparison of the slopes at various temperatures.

3.6 Titanium Alloy Honeycomb Test

A test similar to the one conducted on the René 41 honeycomb has been performed on a titanium alloy honeycomb panel. The minimum temperature point obtained was at 325°F and the maximum average temperature was 1000°F. Again, measurements were taken in both air and a vacuum of about 5×10^{-5} mm Hg. From the previous test (René honeycomb) it was noted that the temperature variation over the surface area of the test section was very small, and it was decided, therefore, to install only one thermocouple on each surface for measuring the temperature gradient across the panel. In addition, two couples were attached to the hot side of the panel, opposite a corner and a side guard heater. These were, as before, used to aid in balancing the guard side and corner heater circuits.

The thermocouples used here were .013 in. diameter chromel-alumel and the technique of attaching them to the surface was the same as described in

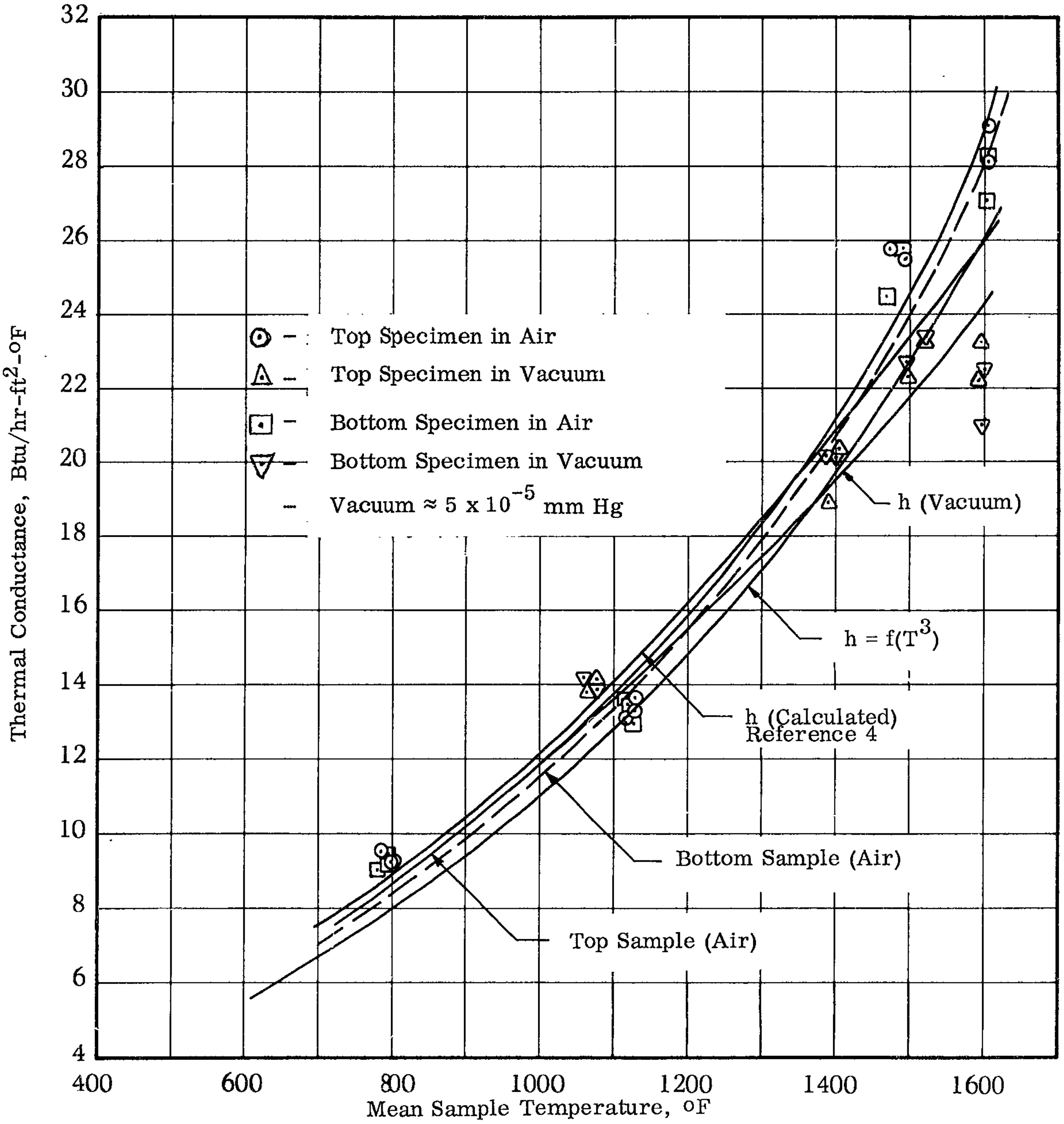


Figure 14. Thermal Conductance of a 0.510"-Thick René 41 Honeycomb Sandwich Panel

Section 3.1. The test panels were 12 in x 12 in x .600 in, with a face sheet thickness of .050 in. The core consisted of square cells, with 1/4 in. cell size, made of .0015 in. foil. The star thermocouple was .004 in. diameter, platinum versus platinum-13%-rhodium, with 16 junctions alternatively on the center section and a guard section of the test specimen.

During the test it was noticed that the thermocouple that had been fastened to the cold side center section of the top sample gave readings completely inconsistent with its equivalent on the bottom sample, all other corresponding temperatures being close to each other (since the over-all thermal resistance of the stack on either side of the main heater is practically the same). As mentioned before, in honeycomb tests the heat transfer to and from the panel occurred by radiation, since the panel was separated from the next components by a small gap. What had happened here was that the cement used to stick the couple junction in the milled slot came loose and under the action of some spring tension in the couple wire, the junction was lifted out of the groove and remained within the gap between the sample and the ratio element. At low temperatures, the temperature gradient across the gap is appreciable and the junction assumed a temperature somewhere between that of the honeycomb cold surface and the surface of the next part, the ratio element in this case. The results as obtained for the bottom sample are recorded in Figure 15.

3.7 High Temperature Honeycomb Test (L 605 Cobalt Base Panel)

A high-temperature, L 605 Cobalt Base, honeycomb panel was tested in both air and vacuum. The maximum average sample temperature was approximately 2100°F. The surface sheets of these honeycomb panels were 0.012 in. thick, while the honeycomb core was fabricated from 0.0015 in. foil. The cell size was 3/16 in and the over-all thickness of the panel, including the surface sheets, was 0.924 in.

Three 0.010 in. wire thermocouples, platinum versus platinum-13%-rhodium, were spot-welded to each face of each sample. These thermocouples were carried out through ceramic insulators. A 0.010 in. wire, platinum-versus-platinum-13%-rhodium star was cemented to the hot face of one of the panels to serve as the star differential.

The data for this honeycomb is presented in Figure 16. There is a considerable difference between the air data which was obtained first and the vacuum data which was run second.

Because of the large decrease in conductivity in the vacuum data compared with the air data, it was decided to run an additional air point following the vacuum test. Although the check point (A) failed to fall back exactly on the first air data, it certainly fell closer to the air than vacuum data. This seems to indicate that the internal convection and gas conduction are substantial with honeycomb structures.

Examination of the honeycomb samples following the test showed considerable oxidation had occurred.

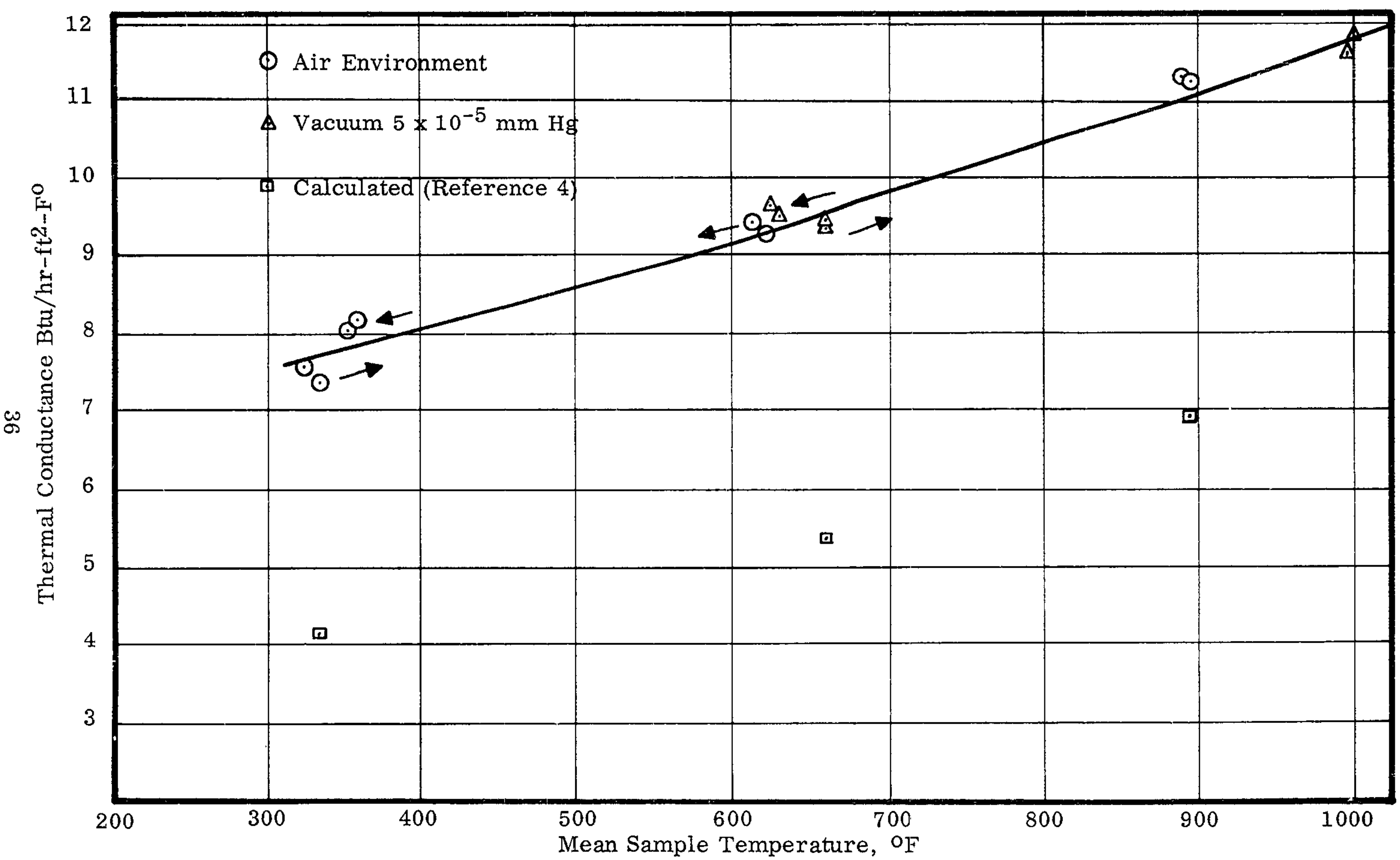


Figure 15. Titanium Alloy Honeycomb Test Results

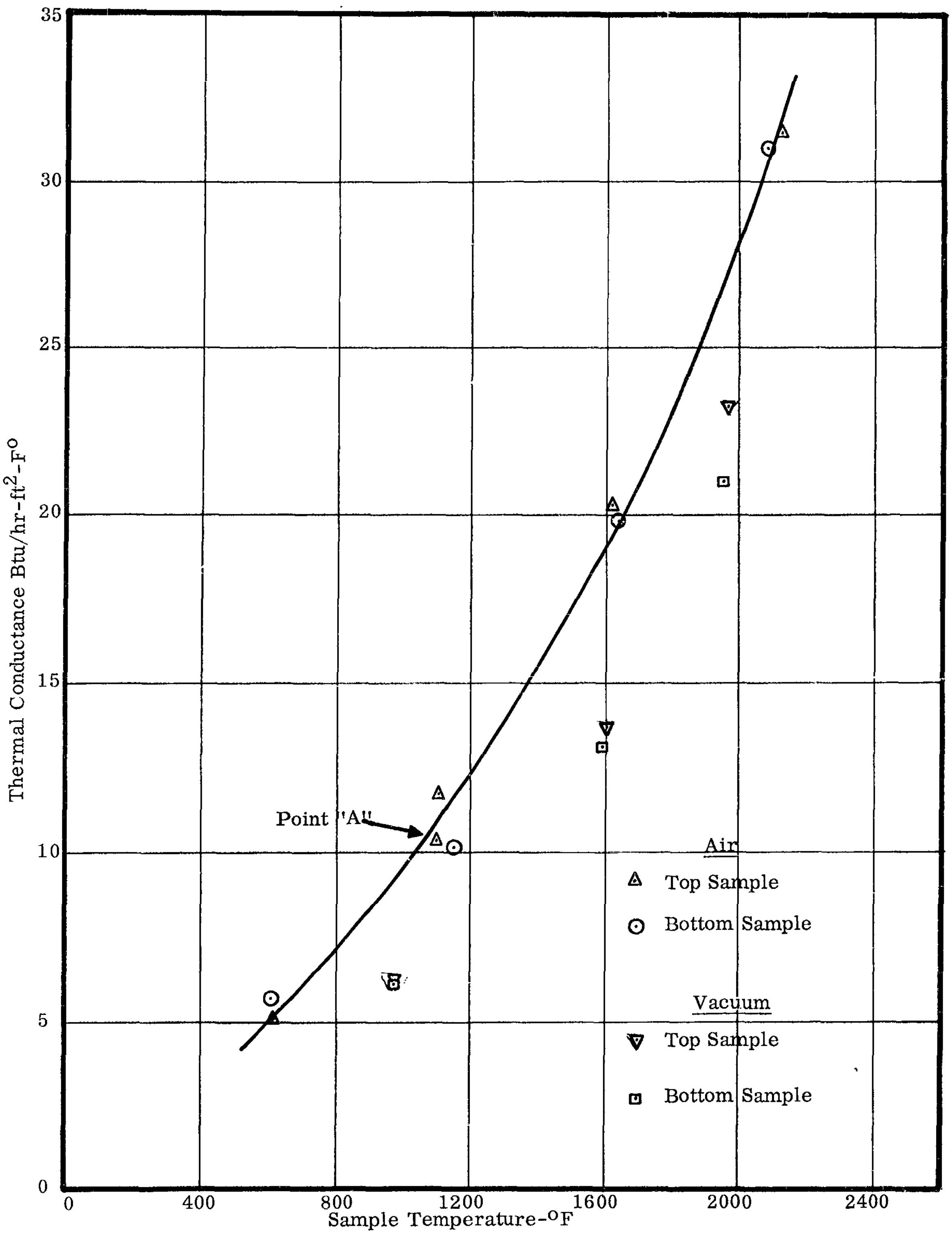


Figure 16. Thermal Conductance vs. Temperature, L-605, Honeycomb

3.8 Precipitation Hardening Stainless Steel Test

As the first of the series of sheet materials to be tested, the thermal conductivity of 17-4PH precipitation hardening stainless steel was measured from around 400°F to 1000°F average sample temperature. The sample consisted of 9 blocks, 4 in x 4 in x 3/4 in. The faces of each block had been ground flat to within ± .002 in. from 3/4 in. Thermocouples, made from .013 in. diameter, bare chromel-alumel wire, enclosed by a 1/16 in. O.D. two-bore ceramic tube, were installed in grooves cut parallel to the surface, sufficiently deep so that the tubes were just level with the surface. The bare junction at the end of the tube was peened into a small, fairly horizontal hole, drilled at the end of the groove. A small correction was made in the thickness of the block, used in the calculation for the thermal conductivity, to account for the fact that the couple junction was below the surface. The star thermocouple was made up of .004 in. diameter platinum-versus-platinum-13%-rhodium wire. As in the copper test (3.3), only one sample was used and an arrangement as described in Section 2.1.2 was made to eliminate heat losses from the top surface of the main heater. The test was run in air first and a few points at the highest temperature have also been obtained in a vacuum of 3 x 10^{-4}mm Hg. The results are plotted in Figure 17, together with a a reference curve by Armco (Reference 5).

3.9 René 41 Test

The test on René 41 solid material was identical with the one conducted on the 17-4PH stainless steel. The thickness of the René 41 blocks was 1/2 in, kept within ± .002 in. by grinding the surfaces. The data have been obtained in the temperature range from 400°F to 1800°F in air as well as in a vacuum of 1 x 10^{-4} mm Hg. The results are plotted in Figure 18. Two curves, obtained by other investigators are recorded for reference.

3.10 Fused Silica Test

The test on a solid insulating material has been performed on fused silica multiform from 400°F to about 2600°F in air and in a vacuum of about 2 x 10^{-5} mm Hg. In each of these environments the thermal conductivity has been determined at several different temperature levels and at each temperature, two sets of data were recorded, approximately one hour apart, to ensure that the apparatus was in a reasonably steady state.

Two samples were used, one on each side of the main heater, and measurements were taken on both. Contrary to previous tests on solid materials, the sample was not cut into nine identical blocks, but kept in one 12 in x 12 in. square slab, 1/2 in. thick. The surface temperatures were measured by means of bare platinum-versus-platinum-13%-rhodium thermocouples, embedded in shallow grooves that had been cut parallel to the surface. To be prepared for thermocouple failure due to silicon attack, and also to obtain an average surface temperature, in case of a slight variation, two couples were installed of different wire size (.010 in. diameter and .020 in. diameter) in each surface. During the tests in air, it was noticed that there were temperature variations over a surface, although not significant.

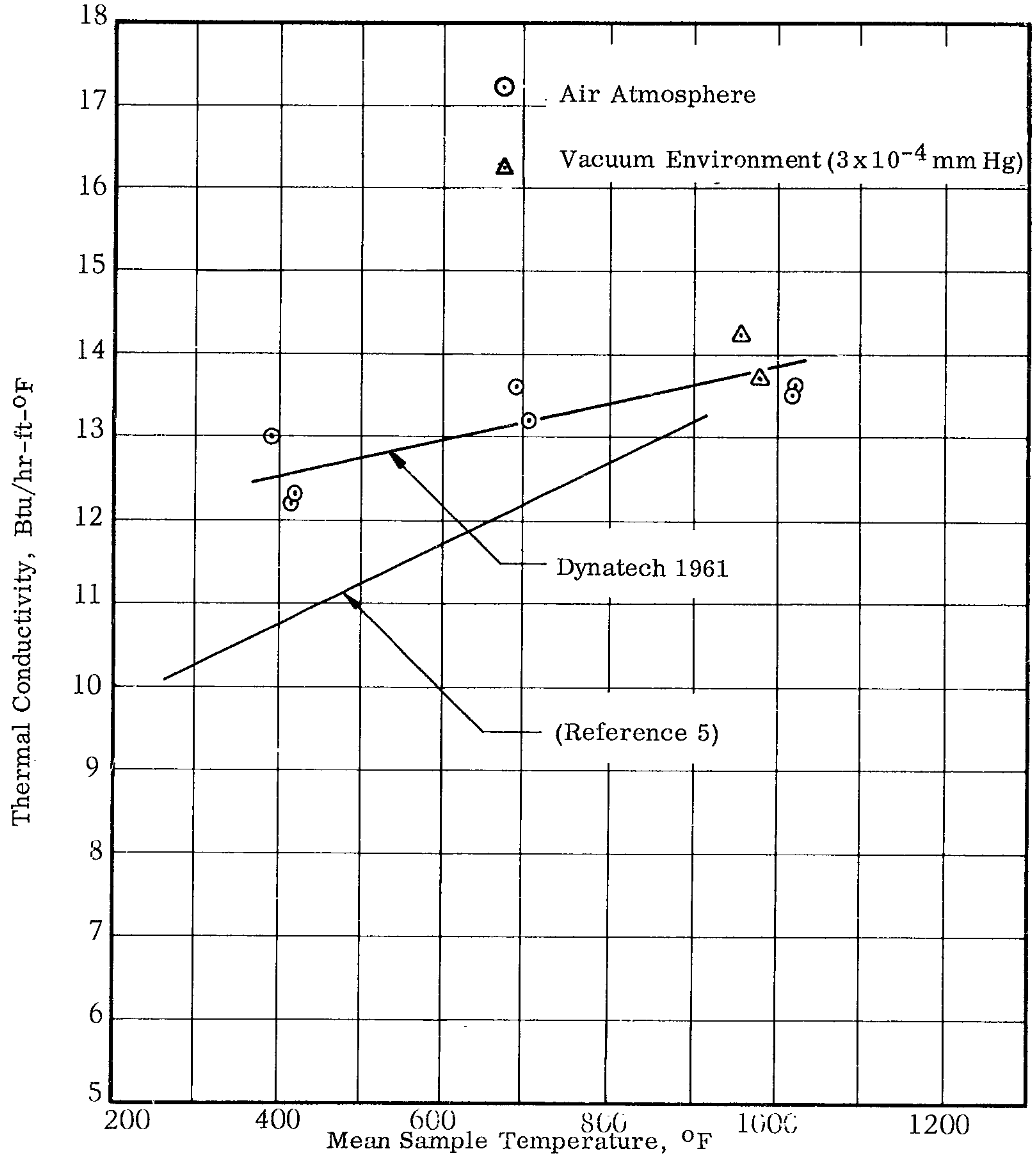


Figure 17. Thermal Conductivity of 17-4 PH Precipitation Hardening Stainless Steel
Fe-19.96 Cr - 4.20 Ni-3.48 Cu
Condition H 900

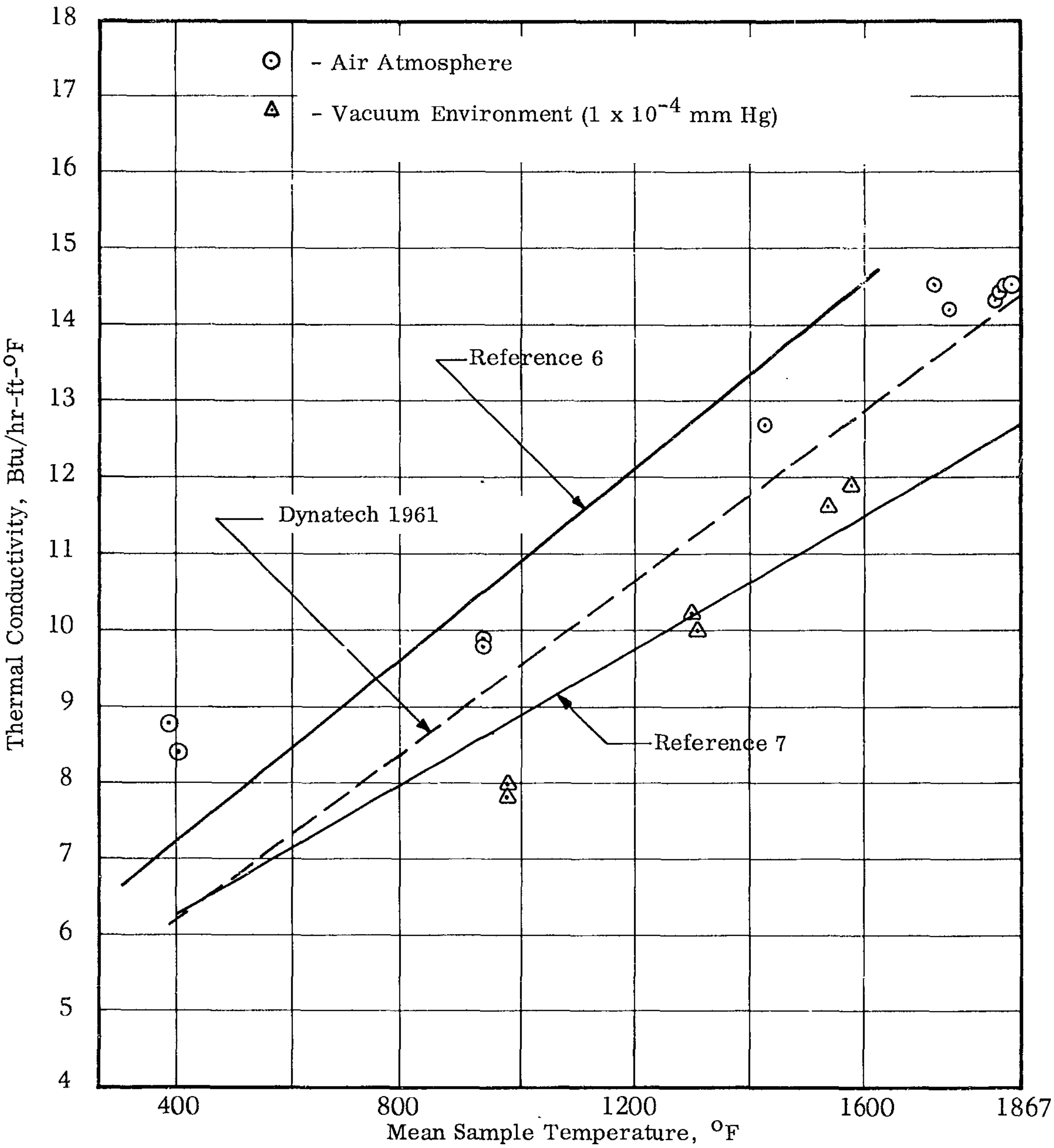


Figure 18. Thermal Conductivity of René 41
N - 19.35 Cr - 11.46 Co - 9.97 Mo - 3.06 Ti - 1.50 Al

The resulting values for the thermal conductivity are shown in the graph together with a few reference data (Figure 19). There is fairly good agreement between the points obtained for each sample at the various temperature levels in air. The sharp rise of the curve at the high temperature is characteristic for quartz materials, as they are transparent to radiation in the lower range of the spectrum. Voids in the material, however, act as scattering centers and may reduce the conductivity again. This could explain the difference between the conductivity as measured during the test and that shown by References 5, 6, and 7 at temperatures in excess of about 2000°F. The test specimen had a porosity of about 8 to 10%.

During the testing in a vacuum, some of the smaller size thermocouples broke, as anticipated, and the reading of only one thermocouple had to be relied on in the determination of the surface temperatures. Because of the resulting reduced accuracy, the data points obtained at each temperature in a vacuum do not coincide as closely as they did in air.

3.11 Tungsten and Columbium Test

It was decided that the tungsten and columbium tests could be performed simultaneously on the apparatus. This would eliminate the necessity of obtaining double samples of each material. Even with a single sample, nine 4 in x 4 in. blocks of each material were required. Thickness of the columbium sample was 1 in. and of the tungsten sample was 1 - 1/2 in. These thicknesses were required to obtain a reasonable temperature difference with the available heat fluxes.

With two dissimilar materials, the ratio elements were to be used to determine the ratio of heat fluxes through the two samples.

Three attempts have been made to perform the tungsten and columbium test all in an argon atmosphere.

During the first test, commercial platinum-sheathed platinum-versus-platinum-rhodium thermocouples, 1/8 in. in diameter, were installed in grooves in each sample. The thermocouples in the surface of one sample were to be used to form the guard balancing differential thermocouple while those in the second sample were used for obtaining data. The roll would then be reversed. Two points were obtained for tungsten at approximately 500°F. These values were 68 and 75 Btu/hr-ft-°F as compared to reference data of 80 Btu/hr-ft-°F. A columbium point was not obtained during this test as the plans were to obtain columbium data on the way down in temperature. This first point was surprisingly accurate considering that in order to obtain a point at 500°F with the auxiliary heaters and ratio elements installed, the heat flux was sufficient to produce a temperature difference of less than 5°F across the sample.

It was expected that as the test progressed to higher temperature the accuracy would become better due to an increase in heat flux and a decrease in thermal conductivity, both of which would produce a larger sample gradient. But as the temperature increased the data became progressively worse. Finally, the test was discontinued and disassembled. It was discovered that almost all of the commercial thermocouples had broken down such that the wires became shorted

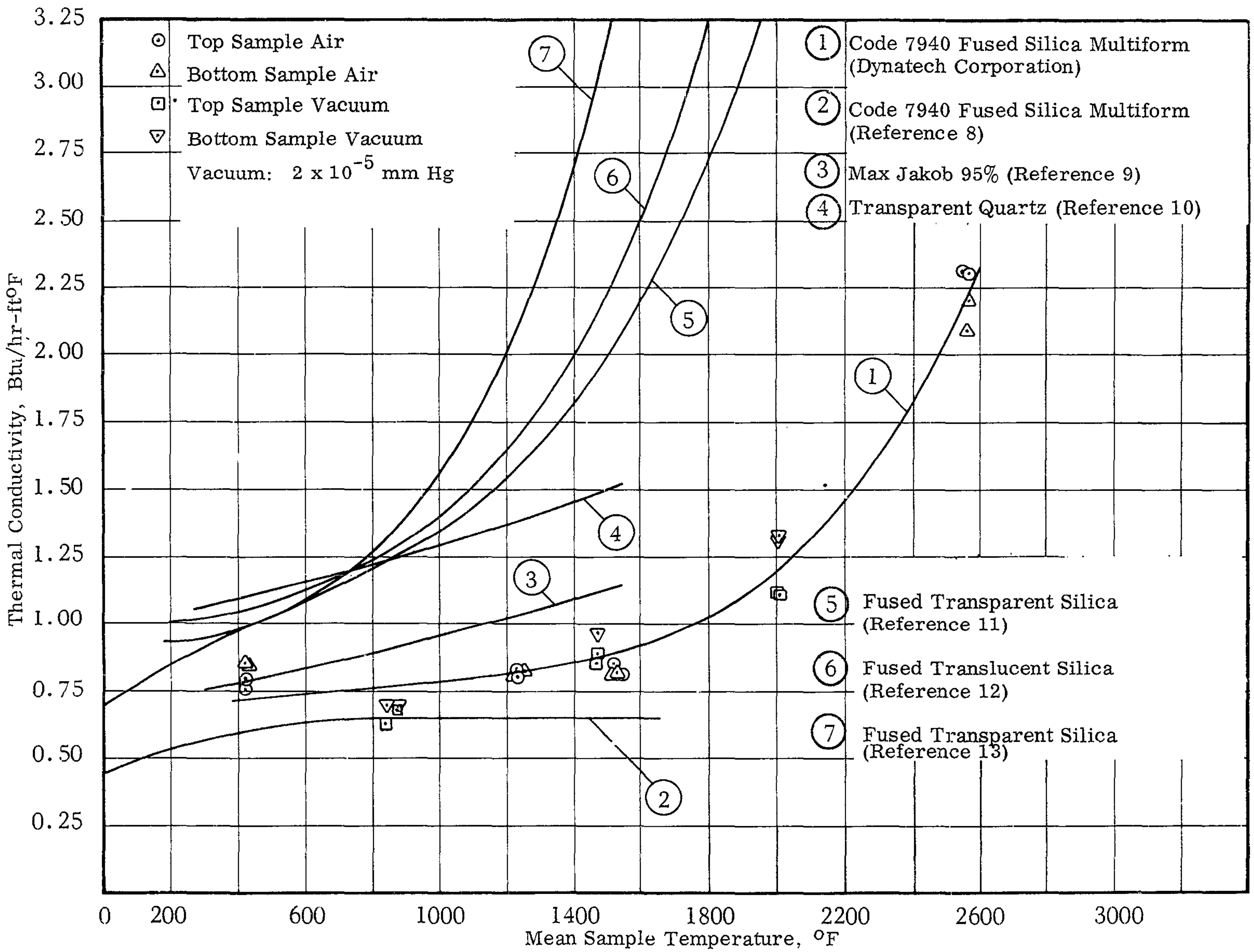


Figure 19. Fused Silica Test Results

at some point in the sheath well before reaching the actual junction. The short was located by passing a soldering iron down the sheath until a sudden potentiometer deflection was obtained.

A second set of commercial thermocouples was obtained, identical to the first in description but from an alternative source. The second tungsten columbium test was performed in the same manner as the first. Two points were obtained for tungsten at an average sample temperature of 1210°F. The value that was obtained for each point was 66 Btu/hr-ft-°F, compared to a reference value of 70.5 Btu/hr-ft-°F. Without attempting to balance the guard heater relative to the columbium sample, a value of 15.3 Btu/hr-ft-°F was obtained at 1270°F compared to a reference value of 31.5 Btu/hr-ft-°F. Gradually the temperature level was increased. The columbium thermocouples began to deteriorate, and at a temperature in excess of 2000°F these thermocouples were lost and several of the heater blocks became discontinuous.

Upon disassembling the stack, a strong attack on the thermocouples and the heater windings was observed. In fact, the thermocouples mounted in the columbium were no longer distinguishable as such. There was just a discolored area in the columbium block where the thermocouples had been.

A new heater was assembled and new thermocouples were installed. This time bare thermocouple wire was run through dense alumina tubes, 1/8 in. O.D., lying in grooves and passing entirely through the test stack into a cold region on either side. During this third test two points were obtained at temperatures of 1200°F and 1800°F. The data was considerably low, explained later by the thermocouple error due to this method of installation as discussed in Section 3.2. Although the thermocouples failed to yield accurate temperatures due to their location, they at least provided consistent values throughout the test, and it is felt that this is an accurate means of protection, at least to 2500°F in tungsten and columbium.

At approximately 2500°F all of the guard heater blocks became discontinuous. The exact failure mechanism is not fully understood as described in Section 3.1. An analysis of the heater wire showed presence of silicon and columbium.

Section 4

CONCLUSIONS

An instrument for measuring the thermal conductance of high temperature structural materials has been designed and developed following the guarded hotplate concept set forth by the ASTM under designation C-177-45 (Reference 1).

Many technical problems, some of which were not originally anticipated, have developed during this program. It is felt that the solutions to these problems, as described in this report have, in some instances, advanced the state of the art. Some of the thermocouple and heater problems and their solutions have not, to the knowledge of the authors, been previously documented.

The idea of applying the guarded hotplate concept to temperatures above the 1400°F recommended by Designation C-177-45, appears to be a sound one as evidenced by the satisfactory copper and aluminum oxide calibration tests to 1800 and 3000°F respectively. This is true provided such problems as material compatibility are not encountered.

The platinum-rhodium heater as supplied by Lexington Laboratories, and modified by Dynatech, is satisfactory to meet the primary objective for the instrument, that is, the testing of high temperature structures of moderate conductivity such as honeycomb configurations. It is not entirely satisfactory to meet all objectives for this instrument however. This heater has failed on each attempt to measure the conductivity of high conductivity refractory metals at high temperatures in vacuum.

With the exception of the tungsten and columbium test, the acceptance check-out and calibration test program has been completed. The experimental work concerning heater elements using resistance wire other than platinum-rhodium to date suggests that a molybdenum-wound heater might work for a single test. The wire becomes extremely brittle during a test and probably will have to be replaced for each successive test.

Section 5

FUTURE PLANS

Dynatech is currently continuing work to develop a heater that will be satisfactory for the remaining tungsten and columbium test. This heater will most probably be an exposed molybdenum-wound alumina block similar in configuration to the present platinum-rhodium heater.

Upon construction of this alternative heater, the final tungsten and columbium test will be completed. With the completion of this final test a supplemental report, describing the alternative heater and the results of the final test, will be issued.

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